

## REMARKS

Claim 23 is amended; support for the amendment is found on page 12, lines 10-21

### Allowable Subject Matter

Applicants sincerely appreciate the Examiner's careful consideration of claims 35, 43 and 44.

### Rejection Under 35 USC § 103 over Piana in View of Tanaka '536.

Claims 23, 24, 26-32, 34, 36, 37, 40, 41, and 45 stand rejected under 35 U.S.C. § 103(a) as unpatentable over Piana, U.S. Patent No. 5,739,204 in view of Tanaka et al., U.S. Patent No. 6,482,536. This rejection is respectfully traversed.

Applicants must respectfully disagree with the Examiner that the combination of Piana and Tanaka anywhere disclose conductive pigments or in any way would serve to make obvious a coating material for the coil coating process comprising a conductive pigment. In view of the Examiner's comments in answer, Applicants have introduced an amendment to provide that the coating composition produces weldable coatings. In further reply to the Examiner's comments, Applicants submit with this response attachments showing that carbon black pigment comes in both conductive and nonconductive grades. Further, carbon black is commonly used in small amounts to provide color, while special grades and sufficient amounts to enable the pigment particles to network to allow conductivity. This situation is obviously not inherent in the bare mention of carbon black in Piana, column 5 "in order to obtain an opaque or pigmented film of the required covering power." Regarding the mention of chromium pigments, the submitted materials show that these are not chromium **metal**, but rather are various chromium **compounds**

such as chromium oxides and chromates. The metal may be electrically conductive, but it is not disclosed as being used in the Piana coating. For anticipation or obviousness to be found by inherency, the property must necessarily be present. Trintec Indus., Inc. v. Top-U.S.A. Corp., 295 F.3d 1292, 1295 (Fed. Cir. 2002) ("Inherent anticipation requires that the missing descriptive material is 'necessarily present,' not merely probably or possibly present, in the prior art." (quoting In re Robertson, 169 F.3d 743, 745 (Fed. Cir. 1999))). To be "necessarily present" the feature must *always* be present in what the prior reference describes. *W.L. Gore & Associates v. Garlock, Inc.*, 220 U.S.P.Q. 303, 314 (Fed. Cir. 1983) (no anticipation where the claimed product had a "unique nature" and the processes of the prior references would not always inherently "produce products meeting all of the claim limitations"). Applicants believe that the claim amendment and the submitted information addresses the point raised by the Examiner.

Accordingly, because the combined references do not in any way lead one to make a weldable coil coating, the person skilled in the art could not know or suspect that the claimed compositions could provide the unique properties arising from Applicants' invention.

In addition, Applicants submit that the properties produced using the claimed compositions are unexpected and further evidence of patentability.

As disclosed on pages 4 to 5, this invention addresses the problem of replacing cathodically deposited primers (cathodic electrodeposition primers) with a coil coating that allows lower baking temperatures, especially for bake hardened steels. It is important for these coil coatings to have extremely high mechanical integrity and to be spot weldable, with a still aesthetically appealing periphery. The coatings also feature a comparatively broad welding range and a comparatively low volume resistant, resulting in a particularly long lifetime for welding electrodes. The claimed coating materials produce coil coatings of extremely high

mechanical integrity and particularly good weldability, especially in spot welding, producing spot welds with a smoother, more aesthetically appealing periphery. Page 7, lines 7-19. The coatings have comparatively broad welding range, low volume resistance, and strong weld connections. Page 7, lines 19-25. The coil coatings also had outstanding mechanical properties, allowing deformation (during shaping) without problems. Page 7, lines 25-28. These properties were obtained at significantly lower PMTs (peak metal temperatures) on curing. Page 8, lines 1-4.

The Examples 1 and 2 (pages 28 to 32) demonstrate that the inventive coating remarkably achieves all these many, different requirements so as to allow replacement of a cathodic electrodeposition primer with a much more economical, environmentally friendly coil coating. Thus, the Example cured at a PMT of 154°C. Page 30, line 25. In contrast, prior art coating have a PMT of 200 to 260°C. Page 1, lines 20-22. The coating had a welding range (kA) of 1.9, a volume resistance of 0.0187 ohms, and allowed 1000 weld spots to be applied using one electrode. Page 31, line 28 to page 32, line 2. In addition, the quality of the weld spots was extremely high. Page 32, lines 2-3. Finally, the coating physical properties were extremely high, and the coating was outstandingly deformable. Page 32, lines 5-20.

Applicants respectfully request withdrawal of the rejection and reconsideration of the claims.

Rejection Under 35 USC § 103 over Piana in View of Tanaka '536 and Smith et al.

Claim 25 stands rejected under 35 U.S.C. § 103(a) as unpatentable over Piana, U.S. Patent No. 5,739,204 in view of Tanaka et al., U.S. Patent No. 6,482,536, and further in view of Smith et al., U.S. Patent No. 5,852,162. This rejection is respectfully traversed.

Applicants submit that claim 25 is patentable over the cited combinations of references for the same reasons discussed in the section about with regard to independent claim 23. The Smith patent is cited as teaching a particular polyester resin. The Smith patent fails, however, to provide the teachings missing from the first two references with regard to underlying independent claim 23, or to explain why such compositions should be further modified, or how such modifications could be expected to successfully produce Applicants' invention.

Thus, for the same reasons, Applicants respectfully request withdrawal of the rejection and reconsideration of claim 25.

Rejection Under 35 USC § 103 over Piana in View of Tanaka '536 and Lorenz et al.

Claim 33 stands rejected under 35 U.S.C. § 103(a) as unpatentable over Piana, U.S. Patent No. 5,739,204 in view of Tanaka et al., U.S. Patent No. 6,482,536, and further in view of Lorenz et al., U.S. Patent Application Publication 2003/0175541. This rejection is respectfully traversed.

The Lorenz publication is cited as disclosing conductive pigment. However, the mere existence of conductive pigment or the Lorenz compositions does not make it obvious to modify the Piana composition in any particular way. In addition, there was no hint in the art that Applicants' particular composition would provide the unexpected benefits outlined above in regard to the rejection of the underlying independent claim.

Accordingly, Applicants respectfully request withdrawal of the rejection and reconsideration of claim 33.

Rejection Under 35 USC § 103 over Piana in View of Tanaka '536 and Rees

Claim 42 stands rejected under 35 U.S.C. § 103(a) as unpatentable over Piana, U.S. Patent No. 5,739,204 in view of Tanaka et al., U.S. Patent No. 6,482,536, and further in view of Rees, U.S. Patent No. 4,826,899. This rejection is respectfully traversed.

The Rees patent is nonanalogous art and is, therefore, unavailable to support an obviousness rejection. To be analogous art, the reference must either be in the field of Applicants' endeavor or be reasonably pertinent to the particular problem that Applicants sought to resolve. *In re Clay*, 23 U.S.P.Q.2d (BNA) 1058, 1060 (Fed. Cir. 1992). See also *Andersen Corp. v. Pella Corp.*, 2008 U.S. App. LEXIS 24087 (Fed. Cir. Nov. 19, 2008) (nonprecedential) (evidence earlier mesh not part of insect screen manufacturing filed and prior art would teach away from using that mesh raises issue on obviousness; further evidence mesh would not meet objective of invention); compare, *In re Lam*, 2002 U.S. App. LEXIS 9704 (reference was analogous art because reasonably pertinent to inventor's problem). The Rees patent concerns low smoking, flame resistant thermoplastics. Title; Abstract. The invention is directed to a composition useful for covering bundles of insulated telecommunication wires. Col. 2, ll. 13-17. The Rees invention seeks to overcome the problem of smoke generation when such compositions are burned. Col. 1, ll. 33-36 & 58-61; col. 2, ll. 19-22. Thus, the Rees patent is neither in the field of Applicant's invention, coil coating compositions, nor does it concern the problem of weldability that Applicants faced.

In response to the Examiner's comments, Applicants point out that the reference must be pertinent to **Applicant's** problem, not something that might be in another cited prior art reference. Moreover, "Piana and Rees clearly teach to use polyester" doesn't mean that Rees is in this field of invention or that Rees addresses a relevant problem.

In addition, there would be not expectation that an additive in the Rees thermoplastic wire covering would behave in any predictable way in the significantly different thermoset coil coatings of the first two references.

Finally, the Rees patent fails to explain or accommodate the deficiencies of the first two references with regard to the features of underlying independent claim 23. Accordingly, for all of these reasons, Applicants continue to believe claim 42 is patentable over the combined references and request reconsideration of claim 42.

#### Rejection Under 35 USC § 103 over Piana in View of Yamada et al.

Claims 23, 24, 27-32, 40, and 45 stand rejected under 35 U.S.C. § 103(a) as unpatentable over Piana, U.S. Patent No. 5,739,204 in view of Yamada et al., U.S. Patent No. 4,734,467. This rejection is respectfully traversed.

Just as in the case of the combination of Piana with the Tanaka patent, the combination of Piana and Tanaka do not anywhere disclose conductive pigments or in any way make obvious a coating material for the coil coating process comprising a conductive pigment with Applicants' disclosed unexpected advantages. The Piana patent does not anywhere disclose or suggest compositions containing electrically conductive pigments. For this reason, each of the claims is patentable over the combined references. In addition, the Examples 1 and 2 (pages 28 to 32) demonstrate that the inventive coating remarkably achieves all the many, different requirements so as to allow replacement of a cathodic electrodeposition primer with a much more economical, environmentally friendly coil coating, a result that could not have been expected from the combined prior art.

Because the combined references do not in any way lead one to make a composition that

will provide a weldable coil coating, as required in the amended claims, the person skilled in the art could not know or suspect that the claimed compositions could provide the unique properties arising from Applicants' invention. Applicants thus request reconsideration of the claims.

Rejection Under 35 USC § 103 over Piana in View of Yamada et al. and Smith et al.

Claim 25 stands rejected under 35 U.S.C. § 103(a) as unpatentable over Piana, U.S. Patent No. 5,739,204 in view of Yamada et al., U.S. Patent No. 4,734,467 and further in view of Smith et al., U.S. Patent No. 5,852,162. This rejection is respectfully traversed.

Applicants submit that claim 25 is patentable over the cited combinations of references for the same reasons discussed in the section about with regard to independent claim 23. The Smith patent is cited as teaching a particular polyester resin. The Smith patent fails, however, to provide the teachings missing from the first two references with regard to underlying independent claim 23, or to explain why such compositions should be further modified, or how such modifications could be expected to successfully produce Applicants' invention.

Thus, for the same reasons, Applicants respectfully request withdrawal of the rejection and reconsideration of claim 25.

Rejection Under 35 USC § 103 over Piana in View of Yamada et al. and Tanaka '003

Claims 26, 34, and 36-39 stand rejected under 35 U.S.C. § 103(a) as unpatentable over Piana, U.S. Patent No. 5,739,204 in view of Yamada et al., U.S. Patent No. 4,734,467 and further in view of Tanaka, U.S. Patent No. 5,623,003. This rejection is respectfully traversed.

Applicants submit that each of claims 6, 34, and 36-39 is patentable over the cited combinations of references for the same reasons discussed in the section about with regard to

independent claim 23. The Tanaka '003 patent is cited as teaching certain features of these dependent claim but it fails to provide the teachings missing from the first two references with regard to underlying independent claim 23, or to explain why such compositions should be further modified, or how such modifications could be expected to successfully produce Applicants' invention.

Thus Applicants respectfully request withdrawal of the rejection and reconsideration of claims 26, 34, and 36-38.

Conclusion

It is believed that a full and complete response has been made to the Office Action, and as such, the present application is in condition for allowance. Thus, prompt and favorable consideration of this amendment is respectfully requested. If the Examiner believes that personal communication will expedite prosecution of this application, the Examiner is invited to telephone the undersigned at (248) 641-1220.

Respectfully submitted,



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US 20060096694A1

(19) **United States**

(12) **Patent Application Publication** (10) **Pub. No.: US 2006/0096694 A1**  
**Zhou** (43) **Pub. Date: May 11, 2006**

(54) **HIGH MODULUS, NONCONDUCTIVE  
ADHESIVE USEFUL FOR INSTALLING  
VEHICLE WINDOWS**

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(21) **Appl. No.: 10/983,387**

(22) **Filed: Nov. 8, 2004**

**Publication Classification**

(51) **Int. Cl.**  
**B32B 17/10 (2006.01)**  
**B60J 1/00 (2006.01)**  
**H01B 1/24 (2006.01)**  
(52) **U.S. Cl. .... 156/99; 156/108; 252/511**

(57) **ABSTRACT**

The invention is a composition comprising

a) one or more isocyanate functional polyether based pre-polymers containing one or more organic based polymers dispersed therein;

b) one or more isocyanate functional polyester based pre-polymers which is solid at 23° C.;

c) one or more polyisocyanates having a nominal functionality of about 3 or greater;

d) one or more conductive carbon blacks in an amount such that the composition has a dielectric constant of about 15 or less; and

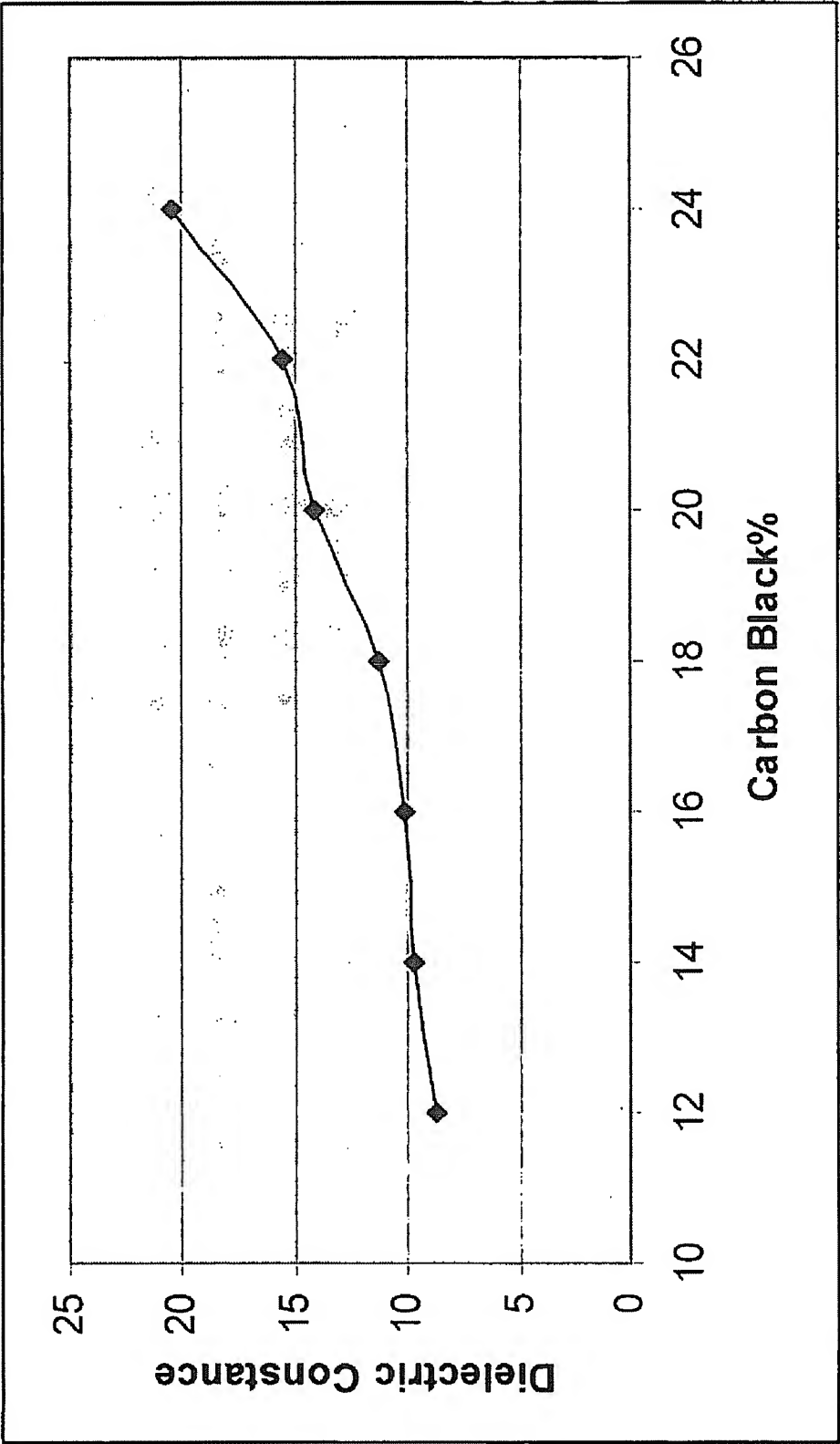
e) one or more catalysts for the reaction of isocyanate moieties with hydroxyl groups,

wherein the composition demonstrates upon cure a modulus of 2.0 MPa or greater at 25° C. measured according to ASTM D4065; a dielectric constant of about 15 or less; a sag of an uncured sample of less than 2 mm, a press flow viscosity of about 20 to about 50 and a storage modulus of about  $5.3 \times 10^5$  Pa or greater.

# [0034]

nonconductive carbon blacks  
carbon black used, but  
composition nonconductive

Figure 1. Dielectric Constance vs. Carbon Black Percent



HIGH MODULUS, NONCONDUCTIVE ADHESIVE USEFUL FOR INSTALLING VEHICLE WINDOWS

# **HIGH MODULUS, NONCONDUCTIVE ADHESIVE USEFUL FOR INSTALLING VEHICLE WINDOWS**

## **FIELD OF INVENTION**

[0001] The invention relates to a composition useful as an adhesive which is useful in bonding glass into vehicles and buildings which adhesive demonstrates high modulus and nonconductive properties. In another embodiment, the invention is a method of bonding two or more substrates together, wherein such substrates may include glass, buildings and vehicles. In another embodiment, the invention is a method of replacing a window in a vehicle using the composition of the invention.

## **BACKGROUND OF INVENTION**

[0002] Adhesive compositions are used to affix (bond) glass (windows) into buildings and vehicles, see Rizk, U.S. Pat. No. 4,780,520; Bhat, U.S. Pat. No. 5,976,305; Hsieh et al, U.S. Pat. No. 6,015,475 and Zhou, U.S. Pat. No. 6,709,539, all incorporated herein by reference. In automobile factories windows are installed using robots and computer controlled processing. This facilitates the use of a variety of high performance adhesives used on a variety of automobiles, for instance nonconductive adhesives and high modulus adhesives. Further, new vehicles are not driven a significant distance for several days after window installation, and thus the speed of cure is not a significant issue. Conversely, when a vehicle needs a window replaced, it is often performed in a remote location by an installer working from a vehicle. In this environment, speed of cure is important as the vehicle owner desires to drive the vehicle away as soon as possible after installation on the window. Adhesives useful in replacing windows for vehicles which facilitate fast drive away times are known see Bhat, U.S. Pat. No. 5,976,305 and Zhou, U.S. Pat. No. 6,709,539. The introduction of various high performance adhesive compositions used for installing windows in automobile factories presents a problem for replacement window installers. First adhesives that meet all the varied performance requirements are not available in the market place. Second, it is difficult to formulate many high performance adhesive compositions to allow rapid drive away times, such as one hour and more preferably 30 minutes. Thus, a replacement window installer often has to carry a variety of adhesives so that the installer can match the adhesive to the properties of the original adhesive. It is also difficult to formulate a high performance adhesive that does not sag, that is, lose the shape of the adhesive bead applied to the vehicle or the glass.

[0003] Adhesives have been developed which provide good initial green strength that allows the adhesive to hold the glass in place without additional fixturing to hold the glass in place. This is achieved through the inclusion of crystalline polyesters in the adhesive. These adhesives have hot melt properties that require that the adhesive be melted and applied hot. As the adhesive cools the polyester portion crystallizes and provides initial green strength to hold the glass in place, see Proebster U.S. Pat. No. 5,747,581, incorporated herein by reference. The problem with these adhesives is that they require heat to apply and the use of complex equipment including a heater for their use. The initial green strength provided is not sufficient for rapid drive away time. In the replacement glass industry segment rapid strength development is necessary to allow safe rapid drive

away times. Because of the use of hot melt adhesives in the automobile window replacement market, many installers insist on heating adhesives prior to applying the adhesive to the window or the window flange. Many adhesives when heated demonstrate sagging, that is the deformation as the result of gravitational forces. This deformation if severe enough can interfere in the proper installation and sealing of the window into the vehicle.

[0004] Several approaches to providing non-conductive adhesives including using non-conductive carbon black in adhesive formulations are known, see commonly assigned patent application Ser. No. 10/921,635 filed Aug. 19, 2004 titled COMPOSITION USEFUL AS AN ADHESIVE FOR INSTALLING VEHICLE WINDOWS. WO 02/053671 discloses the use of low or non-oxidized carbon black with polycarbonate based polyols to achieve this objective. The problem with this technology is that low, conductive carbon black and polycarbonate polyols are significantly more expensive than standard grades of carbon black which are conductive.

[0005] Windows in buildings and vehicles are installed and replaced in a variety of environmental conditions. Many of the present adhesive compositions are limited with respect to the environmental conditions of application. As a result window installers often need to use different adhesives based on the environmental conditions.

[0006] What is needed is a composition which is useful as an adhesive for bonding glass into a structure which exhibits a variety of high performance properties (such as high modulus and nonconductive nature), exhibits fast safe drive away times when applied under a variety of conditions, fast strength development, can be applied without the need for heating the adhesive, can be applied under a wide range of environmental conditions, does not require expensive ingredients and does not sag when applied. Further, what is needed is an adhesive composition which does not sag when heated prior to application to temperatures of up to 80° C.

## **SUMMARY OF INVENTION**

[0007] In one embodiment, the invention is a composition comprising

[0008] a) one or more isocyanate functional polyether based prepolymers containing one or more organic based polymers dispersed therein;

[0009] b) one or more isocyanate functional polyester based prepolymers which is solid at 23° C.;

[0010] c) one or more polyisocyanates having a nominal functionality of about 3 or greater;

[0011] d) one or more conductive carbon blacks in an amount such that the composition has a dielectric constant of about 15 or less; and

[0012] e) one or more catalysts for the reaction of isocyanate moieties with hydroxyl groups, wherein the composition demonstrates upon cure a modulus of about 2.0 MPa or greater at 25° C. measured according to ASTM D4065; a dielectric constant of about 15 or less; a sag of an uncured sample of less than about 2 mm, a press flow viscosity of about 20 to about 50 and a storage modulus of about  $5.3 \times 10^5$  Pa or greater.

[0013] In another embodiment the invention is a method of bonding two or more substrates together which comprises contacting the two or more substrates together with a composition according to this invention disposed along at least a portion of the area wherein the substrates are in contact.

[0014] In yet another embodiment the invention is a method of replacing a window of a vehicle comprising

[0015] i) removing the window from the vehicle;

[0016] ii) applying a composition according to the invention to a replacement window or to the flange of the vehicle adapted to hold the window into the vehicle;

[0017] iii) contacting the flange of the vehicle and the replacement window with the composition disclosed between the replacement window and the flange of the vehicle; and

[0018] iv) allowing the adhesive to cure.

[0019] The composition of the invention is useful as an adhesive to bond substrates together. A variety of substrates may be bonded together using the composition, for instance, plastics, glass, wood, ceramics, metal, coated substrates, such as plastics with an abrasion resistant coating disposed thereon, and the like. The compositions of the invention may be used to bond similar and dissimilar substrates together. The compositions are especially useful for bonding glass or a plastic with an abrasion resistant coating disposed thereon to other substrates such as vehicles and buildings. The compositions of the invention are also useful in bonding parts of modular components together, such as vehicle modular components. The glass or plastic with an abrasion resistant coating disposed thereon can be bonded to coated and uncoated portions of vehicles. Advantageously the adhesive is pumpable, sag resistant and functional, bonds parts together, at temperatures between about 20° C. and about 80° C. This allows the adhesives prepared from the composition of the invention to be applied at a wide range of ambient temperatures. Heated application machinery is not necessary for the application of the adhesive. Furthermore, the adhesive demonstrates rapid strength development which facilitates rapid drive away times of preferably one hour, and more preferably 30 minutes, after application of the adhesive at temperatures of from about 0° F. (-18° C.) to about 115° F. (46° C.). In particular, windshields installed under such conditions meet United States Federal Motor Vehicle Safety Standard (FMVSS) 212. The compositions of the invention are nonconductive and demonstrate a dielectric constant of about 15 or less. The compositions of the invention preferably demonstrate a modulus after application for two weeks of about 2 MPa or greater, more preferably about 2.2 MPa or greater and preferably about 3 MPa or less according to ASTM D4065. The compositions of the invention exhibit a storage modulus,  $G'$ , of about  $5.3 \times 10^8$  Pa or greater, preferably about 0.53 MPa or greater and most preferably about 1.0 MPa or greater. Pumpability of the composition can be measured according to the press flow viscosity test described hereinafter; according to that test the composition exhibits a press flow viscosity of about 20 to about 50 seconds.

#### DESCRIPTION OF FIGURES

[0020] FIG. 1 is a graph of conductive carbon percentage versus dielectric constant.

#### DETAILED DESCRIPTION OF INVENTION

[0021] One or more as used herein means that at least one, or more than one, of the recited components may be used as disclosed. Nominal as used with respect to functionality means the theoretical functionality, generally this can be calculated from the stoichiometry of the ingredients used. Generally, the actual functionality is different due to imperfections in raw material, incomplete conversion of the reactants and formation of by-products.

[0022] The one or more isocyanate functional polyether based prepolymers are present in sufficient quantity to provide adhesive character to the composition. Such prepolymers have an average isocyanate functionality sufficient to allow the preparation of a crosslinked polyurethane upon cure and not so high that the polymers are unstable. Stability in this context means that the prepolymer or adhesive prepared from the prepolymer has a shelf life of at least 6 months at ambient temperatures, in that it does not demonstrate an increase in viscosity during such period which prevents its application or use. Preferably the prepolymer or adhesive prepared therefrom does not undergo an increase in viscosity of more than about 50 percent during the stated period. The prepolymer preferably has a free isocyanate content, which facilitates acceptable strength in adhesives prepared from the prepolymers after 60 minutes and stability of the prepolymer. Preferably, the free isocyanate content is about 0.8 percent by weight or greater based on the weight of the prepolymer and more preferably about 0.9 percent by weight or greater, and preferably about 2.2 percent by weight or less, more preferably about 2.0 or less, even more preferably about 1.4 percent by weight or less and even more preferably about 1.1 percent by weight or less and most preferably about 1.0 percent by weight or less. Above about 2.2 percent by weight the adhesives prepared from the prepolymer may demonstrate lap shear strengths after 60 minutes which are too low for the intended use. Below about 0.8 percent by weight the prepolymer viscosity is too high to handle and the working time is too short.

[0023] The prepolymer preferably exhibits a viscosity, which facilitates formulation of a pumpable adhesive which has good green strength. Preferably the viscosity of the prepolymer is about 100,000 centipoise (100 Pa s) or less and more preferably about 70,000 centipoise (70 Pa s) or less, and most preferably about 45,000 centipoise (45 Pa s) or less and about 30,000 centipoise (30 Pa s) or greater. The viscosity used herein is Brookfield viscosity determined using a number 5 spindle. The viscosity of the adhesive can be adjusted with fillers, although the fillers generally do not improve the green strength of the final adhesive. Below about 30,000 centipoise (30 Pa s) the adhesive prepared from the prepolymer may exhibit poor green strength. Above about 100,000 (100 Pa s) the prepolymer may be unstable and hard to dispense. The prepolymer may be prepared by any suitable method, such as by reacting polyols, such as diols, triols and dispersion triols such as a copolymer polyol or grafted triol, with an excess over stoichiometry of one or more polyisocyanates under reaction conditions sufficient to form a prepolymer having isocyanate functionality and free isocyanate content which meets the criteria discussed above. In a preferable method used to prepare the prepolymer, the polyisocyanates are reacted with one or more diols, one or more triols and one or more dispersion triols.

[0024] Preferable polyisocyanates for use in preparing the prepolymer include those disclosed in U.S. Pat. No. 5,922,809 at column 3, line 32 to column 4, line 24 incorporated herein by reference. Preferably the polyisocyanate is an aromatic or cycloaliphatic polyisocyanate such as diphenylmethane-4,4'-diisocyanate, isophorone diisocyanate, tetramethylxylene diisocyanate, and is most preferably diphenylmethane-4,4'-diisocyanate. The diols and triols are generically referred to as polyols. Polyols useful in this invention are diols and triols corresponding to the polyols described in U.S. Pat. No. 5,922,809 at column 4, line 60 to column 5, line 50, incorporated herein by reference. Preferably the polyols (diols and triols) are polyether polyols and more preferably polyoxyalkylene oxide polyols. Most preferred triols are ethylene oxide-capped polyols prepared by reacting glycerin with propylene oxide, followed by reacting the product with ethylene oxide.

[0025] Preferably the prepolymer contains one or more organic based polymers dispersed therein. Preferably the organic based polymer is included in the prepolymer by inclusion of a dispersion triol having dispersed therein particles of an organic based polymer. The preferable dispersion triols are disclosed in Zhou, U.S. Pat. No. 6,709,539 at column 4, line 13 to column 6, line 18, incorporated herein by reference. Preferably the triol used to disperse the organic particles is a polyether triol and more preferably a polyoxyalkylene based triol. Preferably, such polyoxyalkylene oxide triol comprises a polyoxypropylene chain with a polyoxyethylene end cap. Preferably the triols used have a molecular weight of about 4,000 or greater, more preferably about 5,000 or greater and most preferably about 6,000 or greater. Preferably such triol has molecular weight of about 8,000 or less and more preferably about 7,000 or less.

[0026] Preferably the particles dispersed in the dispersion triol comprise a thermoplastic polymer, rubber-modified thermoplastic polymer or a polyurea dispersed in a triol. The polyurea preferably comprises the reaction product of a polyamine and a polyisocyanate. Preferable thermoplastic polymers are those based on monovinylidene aromatic monomers and copolymers of monovinylidene aromatic monomers with conjugated dienes, acrylates, methacrylates, unsaturated nitrites or mixtures thereof. The copolymers can be block or random copolymers. More preferably the particles dispersed in the triol comprise copolymers of unsaturated nitrites, conjugated dienes and a monovinylidene aromatic monomer, a copolymer of an unsaturated nitrile and a monovinylidene aromatic monomer or a polyurea. Even more preferably the particles comprise a polyurea or polystyrene-acrylonitrile copolymer with the polystyrene-acrylonitrile copolymers being most preferred. The organic polymer particles dispersed in the triol preferably have a particle size which is large enough to improve the impact properties and elastomeric properties of the finally cured adhesive, but not so large so as to reduce the ultimate strength of the adhesive after cure. The particles may be dispersed in the triol or grafted to the backbone of some of the triols. Preferably the particle size is about 10 microns or greater and more preferably the particle size is about 20 microns or greater. Preferably the particle size is about 50 microns or less and more preferably the particle size is about 40 microns or less. The triol dispersion contains a sufficient amount of organic polymer particles such that the adhesive upon cure has sufficient hardness for the desired use and not so much such that the cured adhesive has too much elasticity

as defined by elongation. Preferably the dispersion contains about 20 percent by weight or greater of organic polymer particles copolymer based on the dispersion, preferably about 30 percent by weight or greater and more preferably about 35 percent by weight or greater. Preferably the dispersion contains about 60 percent by weight or less of organic polymer particles based on the dispersion, preferably about 50 percent by weight or less and more preferably about 45 percent by weight or less.

[0027] The polyols (diols and triols and dispersion triols) are present in an amount sufficient to react with most of the isocyanate groups of the isocyanates leaving enough isocyanate groups to correspond with the desired free isocyanate content of the prepolymer. Preferably the polyols are present in an amount of about 30 percent by weight or greater based on the prepolymer, more preferably about 40 percent by weight or greater and most preferably about 55 percent by weight or greater. Preferably the polyols are present in an amount of about 75 percent by weight or less based on the prepolymer, more preferably about 65 percent by weight or less and most preferably about 60 percent by weight or less.

[0028] The weight ratio of diols to triols and dispersion triols is important to achieving the desired cure rate and strength of the adhesive. If the weight ratio is too low the formulation is too viscous to handle and the resulting adhesive has insufficient elasticity to retain glass in an automobile window frame under crash conditions. If the ratio is too high the adhesive does not have adequate green strength. The weight ratio of diol to triol and dispersion triol is preferably about 0.8 or greater and more preferably about 0.85 or greater and most preferably about 0.9 or greater. The weight ratio of diol to triol and dispersion triol is about 1.2 or less; more preferably about 1.0 or less and most preferably about 0.95 or less. In the embodiment where the polyols comprise a mixture of diols and triols, the amount of diols present is preferably about 15 percent by weight or greater based on the prepolymer, more preferably about 25 percent by weight or greater and most preferably about 28 percent by weight or greater; and about 40 percent by weight or less based on the prepolymer, more preferably about 35 percent by weight or less and most preferably about 30 percent by weight or less. In the embodiment where the polyols comprise a mixture of diols and triols, the amount of triols (non dispersion triol and dispersion triol) present is preferably about 15 percent by weight or greater based on the prepolymer, more preferably about 25 percent by weight or greater and most preferably about 28 percent by weight or greater; and preferably about 45 percent by weight or less based on the prepolymer, more preferably about 35 percent by weight or less and most preferably about 32 percent by weight or less.

[0029] The dispersion of organic polymer particles in a triol is present in the prepolymer in an amount of about 10 percent by weight or greater of the prepolymer and more preferably about 12 percent by weight or greater, and about 18 percent by weight or less of the prepolymer and more preferably about 15 percent by weight or less.

[0030] The polyurethane prepolymers of the invention may further comprise a plasticizer. The plasticizers useful in the prepolymer are common plasticizers useful in polyurethane adhesive applications and well known to those skilled in the art. The plasticizer is present in an amount sufficient

to disperse the prepolymer in the final adhesive composition. The plasticizer can be added to the adhesive either during preparation of the prepolymer or during compounding of the adhesive composition. Preferably the plasticizer is present in about 1 percent by weight or greater of the prepolymer formulation (prepolymer plus plasticizer), more preferably about 20 percent by weight or greater and most preferably about 30 percent by weight or greater. Preferably the plasticizer is present in about 45 percent by weight or less of the prepolymer formulation and more preferably about 35 percent by weight or less.

[0031] The polyurethane prepolymer may be prepared by any suitable method, such as bulk polymerization and solution polymerization. Preferable processes for the preparation of the prepolymers are disclosed in U.S. Pat. No. 5,922,809 at column 9, line 4 to 51 incorporated herein by reference. The polyurethane prepolymers are present in the adhesive composition in an amount sufficient such that when the resulting adhesive cures substrates are bound together. Preferably the polyurethane prepolymers are present in an amount of about 25 parts by weight of the adhesive composition or greater, more preferably about 30 parts by weight or greater and most preferably about 35 parts by weight or greater. Preferably, the polyurethane prepolymers are present in an amount of about 55 parts by weight of the adhesive composition or less, more preferably about 50 parts by weight or less and even more preferably about 45 parts by weight or less.

[0032] The composition further comprises one or more isocyanate functional prepolymers containing one or more polyester based polyols which are solid at ambient temperature, about 23° C. The polyester based polyols have melting points such that the prepolymer provides sufficient green strength to prevent the substrates from moving in relation to one another due to gravitational forces and such that the prepolymer is solid at ambient temperatures. In terms of installing a window in a vehicle or building, the polyester based prepolymer prevents the window from sliding after installation. Preferably, the polyester polyols have melting points of about 40° C. or greater, even more preferably about 45° C. or greater and most preferably about 50° C. or greater. Preferably, the polyester polyols exhibit melting points of about 85° C. or less and most preferably about 60° C. or less and even more preferably about 70° C. or less. The polyester based isocyanate prepolymer can be prepared using one or more polyester polyols. The amount of polyester polyol in the prepolymer is a sufficient amount to provide the needed green strength to the composition of the invention and to render it solid. Preferably, the polyester polyol is present in the polyester polyol based isocyanate prepolymer in an amount of about 70 percent by weight or greater based on the weight of the prepolymer and more preferably about 80 percent by weight or greater. Preferably, the polyester polyol is present in the polyester polyol based isocyanate prepolymer in an amount of about 95 percent by weight or less based on the weight of the prepolymer and more preferably about 90 percent by weight or less. Preferably, the polyester polyol based isocyanate prepolymer is present in the adhesive composition in sufficient amount to give the needed green strength and the desired rheology of the composition. Preferably, the polyester polyol based isocyanate prepolymer is present in the adhesive composition in an amount of about 1 percent by weight or greater based on the weight of the prepolymer and most preferably about 2 percent by weight

or greater. Preferably, the polyester polyol based isocyanate prepolymer is present in the adhesive composition in an amount of about 5 percent by weight or less and most preferably about 3 percent by weight or less. The polyester polyol can be any polyester composition that meets the property requirements defined, which is crystalline at ambient temperatures and melts in the desired temperature range. Preferred polyester polyols are prepared from linear diacids and linear diols. A more preferred diacid is adipic acid. More preferred diols are the C<sub>2-6</sub> diols, with butane diols, pentane diols and hexane diols being most preferred. The polyester based polyisocyanate prepolymers can be prepared using the processes and isocyanates described hereinbefore. Preferred polyester polyols are available from Creanova under the trade name Dynacol and the designations 7360 and 7330, with 7360 more preferred.

[0033] The composition of the invention further comprises a polyfunctional isocyanate for the purpose of improving the modulus of the composition in the cured form. Polyfunctional as used in the context of the isocyanates refers to isocyanates having a functionality of 3 or greater. The polyisocyanates can be any monomeric, oligomeric or polymeric isocyanate having a nominal functionality of about 3 or greater. More preferably the polyfunctional isocyanate has a nominal functionality of about 3.2 or greater. Preferably the polyfunctional isocyanate has a nominal functionality of about 5 or less, even more preferably about 4.5 or less and most preferably about 4.2 or less. The polyfunctional isocyanate can be any isocyanate which is reactive with the isocyanate polyisocyanate prepolymers used in the composition and which improves the modulus of the cured composition. The polyisocyanates can be monomeric; trimers, isocyanurates or biurets of monomeric isocyanates; oligomeric or polymeric, the reaction product of several units of one or more monomeric isocyanates. Examples of preferred polyfunctional isocyanates include trimers of hexamethylene diisocyanate, available from Bayer under the trademark and designation Desmodur® N3300, and polymeric isocyanates such as polymeric MDI (methylene diphenyl diisocyanates) such as those marketed by The Dow Chemical Company under the trademark of PAPI™ including PAPI™ 20 polymeric isocyanate. The polyfunctional isocyanates are present in sufficient amount to impact the modulus of the cured compositions of the invention. If too much is used the cure rate of the composition is unacceptably slowed down. If too little is used the desired modulus levels are not achievable. The polyfunctional isocyanate is preferably present in an amount of about 0.5 percent by weight or greater based on the weight of the composition, more preferably about 1.0 percent by weight or greater and most preferably about 1.4 percent by weight or greater. The polyfunctional isocyanate is preferably present in an amount of about 8 percent by weight or less, based on the weight of the composition, more preferably about 5 percent by weight or less and most preferably about 2.5 percent by weight or less.

[0034] The composition of the invention also comprises carbon black to give the composition the desired black color, viscosity and sag resistance. One or more carbon blacks may be used in the composition. The carbon black used in this invention may be a standard carbon black which is not specially treated to render it nonconductive. Standard carbon black is carbon black which is not specifically surface treated or oxidized. One or more nonconductive carbon

blacks may be used in conjunction with the standard carbon black, although such inclusion may add unnecessary costs. The amount of standard carbon black in the composition is that amount which provides the desired color, viscosity, sag resistance and provided the composition is nonconductive to the level defined herein. The standard carbon black is preferably used in the amount of about 10 percent by weight or greater based on the weight of the composition, more preferably about 12 percent by weight or greater and most preferably about 14 percent by weight or greater. The standard carbon black is preferably present in an amount of about 20 percent by weight or less based on the weight of the composition, more preferably about 18 percent by weight or less and most preferably about 16 percent by weight or less. The total carbon black present including conductive or standard and non-conductive carbon black is preferably about 35 percent by weight or less based on the weight of the composition, more preferably about 30 percent by weight or less and most preferably about 20 percent by weight or less. Standard carbon blacks are well known in the art and include Raven™ 790, Raven™ 450, Raven™ 500, Raven™ 430, Raven™ 420 and Raven™ 410 carbon blacks available from Colombian and CSX™ carbon blacks available from Cabot, and Printex™30 carbon black available from Degussa. Non-conductive carbon blacks are well known in the art and include Raven™ 1040 and Raven™ 1060 carbon black available from Colombian.

[0035] The adhesive composition of the invention may further comprise a catalyst known for promoting the cure of polyurethanes in the presence of moisture. Preferable catalysts include metal salts such as tin carboxylates, organo titanates (such as alkyl titanates), metal carboxylates, tertiary amines, such as dimorpholinodiethyl ether or alkyl-substituted dimorpholinodiethyl ethers. Preferably, the catalyst comprises a mixture of metal carboxylates and one of dimorpholino-diethyl ether or an alkyl substituted dimorpholino diethyl ether. Preferred metal carboxylates include bismuth carboxylates. Among preferred catalysts are bismuth octoate, dimorpholinodiethyl ether and (di-(2-(3,5-dimethylmorpholino) ethyl)) ether. Such catalysts, when employed are preferably employed in an amount based on the weight of the adhesive composition of about 0 parts by weight or greater, more preferably about 0.1 parts by weight or greater, even more preferably about 0.2 parts by weight or greater and most preferably about 0.4 parts by weight or greater. Such catalysts are preferably employed in an amount, based on the weight of the adhesive composition of about 5 parts by weight or less, more preferably about 1.75 parts by weight or less, even more preferably about 1 part by weight or less and most preferably about 0.6 parts by weight or less.

[0036] The adhesive of the invention may be formulated with fillers and additives known in the prior art for use in adhesive compositions. By the addition of such materials physical properties such as viscosity flow rates and the like can be modified. However, to prevent premature hydrolysis of the moisture sensitive groups of the polyurethane prepolymer, fillers should be thoroughly dried before admixture therewith.

[0037] Optional components of the adhesive of the invention include reinforcing fillers. Such fillers are well known to those skilled in the art and include carbon black, titanium dioxide, calcium carbonate, surface treated silicas, titanium

oxide, fume silica, talc, and the like. Preferred reinforcing fillers comprise carbon black as described hereinbefore. In one embodiment, more than one reinforcing filler may be used, of which one is carbon black. The reinforcing fillers are used in sufficient amount to increase the strength of the adhesive and to provide thixotropic properties to the adhesive.

[0038] Among optional materials in the adhesive composition are clays. Preferred clays useful in the invention include kaolin, surface treated kaolin, calcined kaolin, aluminum silicates and surface treated anhydrous aluminum silicates. The clays can be used in any form, which facilitates formulation of a pumpable adhesive. Preferably the clay is in the form of pulverized powder, spray-dried beads or finely ground particles. Clays may be used in an amount of about 0 parts by weight of the adhesive composition or greater, more preferably about 1 part by weight or greater and even more preferably about 6 parts by weight or greater. Preferably the clays are used in an amount of about 25 parts by weight or less of the adhesive composition and more preferably about 10 parts by weight or less.

[0039] The adhesive composition of this invention may further comprise plasticizers so as to modify the rheological properties to a desired consistency. Such materials should be free of water, inert to isocyanate groups and compatible with a polymer. Suitable plasticizers are well known in the art and preferable plasticizers include alkyl phthalates such as dioctylphthalate or dibutylphthalate, partially hydrogenated terpene commercially available as "HB-40", trioctyl phosphate, epoxy plasticizers, toluene-sulfamide, chloroparaffins, adipic acid esters, castor oil, toluene and alkyl naphthalenes. The amount of plasticizer in the adhesive composition is that amount which gives the desired rheological properties and which is sufficient to disperse the catalyst in the system. The amounts disclosed herein include those amounts added during preparation of the prepolymer and during compounding of the adhesive. Preferably plasticizers are used in the adhesive composition in an amount of about 0 parts by weight or greater based on the weight of the adhesive composition, more preferably about 5 parts by weight or greater, more preferably about 10 parts by weight or greater and most preferably about 15 parts by weight or greater. The plasticizer is preferably used in an amount of about 40 parts by weight or less based on the total amount of the adhesive composition, more preferably about 30 parts by weight or less and most preferably about 25 parts by weight or less.

[0040] The composition of this invention may further comprise stabilizers, which function to protect the adhesive composition from moisture, thereby inhibiting advancement and preventing premature crosslinking of the isocyanates in the adhesive formulation. Stabilizers known to the skilled artisan for moisture curing adhesives may be used preferably herein. Included among such stabilizers are diethylmalonate, alkylphenol alkylates, paratoluene sulfonic isocyanates, benzoyl chloride and orthoalkyl formates. Such stabilizers are preferably used in an amount of about 0.1 parts by weight or greater based on the total weight of the adhesive composition, preferably about 0.5 parts by weight or greater and more preferably about 0.8 parts by weight or greater. Such stabilizers are used in an amount of about 5.0 parts by weight or less based on the weight of the adhesive compo-



sition, more preferably about 2.0 parts by weight or less and most preferably about 1.4 parts by weight or less.

[0041] The composition of this invention may further comprise an adhesion promoter, such as those disclosed in Mahdi, US Patent Publication 2002/0100550 paragraphs 0055 to 0065 and Hsieh, U.S. Pat. No. 6,015,475 column 5 line 27 to Column 6, line 41 incorporated herein by reference. The amounts of such adhesion promoters useful are also disclosed in these references and incorporated herein by reference.

[0042] The adhesive composition may further comprise a hydrophilic material that functions to draw atmospheric moisture into the composition. This material enhances the cure speed of the formulation by drawing atmospheric moisture to the composition. Preferably, the hydrophilic material is a liquid. Among preferred hydroscopic materials are pyrrolidinones such as 1 methyl-2-pyrrolidinone, available from under the trademark m-pyrol. The hydrophilic material is preferably present in an amount of about 0.1 percent by weight or greater and more preferably about 0.3 percent by weight or greater and preferably about 1.0 percent by weight or less and most preferably about 0.6 percent by weight or less. Optionally the adhesive composition may further comprise a thixotrope. Such thixotropes are well known to those skilled in the art and include alumina, limestone, talc, zinc oxides, sulfur oxides, calcium carbonate, perlite, slate flour, salt (NaCl), cyclodextrin and the like. The thixotrope may be added to the adhesive of composition in a sufficient amount to give the desired rheological properties. Preferably, the thixotrope is present in an amount of about 0 parts by weight or greater based on the weight of the adhesive composition, preferably about 1 part by weight or greater. Preferably, the optional thixotrope is present in an amount of about 10 parts by weight or less based on the weight of the adhesive composition and more preferably about 2 parts by weight or less.

[0043] Other components commonly used in adhesive compositions may be used in the adhesive composition of this invention. Such materials are well known to those skilled in the art and may include ultraviolet stabilizers and antioxidants and the like.

[0044] As used herein all parts by weight relative to the components of the adhesive composition are based on 100 total parts by weight of the adhesive composition.

[0045] The adhesive composition of this invention may be formulated by blending the components together using means well known in the art. Generally, the components are blended in a suitable mixer. Such blending is preferably conducted in an inert atmosphere in the absence of oxygen and atmospheric moisture to prevent premature reaction. It may be advantageous to add any plasticizers to the reaction mixture for preparing the isocyanate containing prepolymer so that such mixture may be easily mixed and handled. Alternatively, the plasticizers can be added during blending of all the components. Once the adhesive composition is formulated, it is packaged in a suitable container such that it is protected from atmospheric moisture and oxygen. Contact with atmospheric moisture and oxygen could result in premature crosslinking of the polyurethane prepolymer-containing isocyanate groups.

[0046] The adhesive composition of the invention is used to bond a variety of substrates together as described here-

inbefore. The composition can be used to bond porous and nonporous substrates together. The adhesive composition is applied to a substrate and the adhesive on the first substrate is thereafter contacted with a second substrate. In preferred embodiments, the surfaces to which the adhesive is applied are cleaned and primed prior to application, see for example, U.S. Pat. Nos. 4,525,511; 3,707,521 and 3,779,794; relevant parts of all are incorporated herein by reference. Generally the adhesives of the invention are applied at ambient temperature in the presence of atmospheric moisture. Exposure to atmospheric moisture is sufficient to result in curing of the adhesive. Curing can be accelerated by the addition of additional water or by applying heat to the curing adhesive by means of convection heat, microwave heating and the like. Preferably the adhesive of the invention is formulated to provide a working time of about 6 minutes or greater more preferably about 10 minutes or greater. Preferably the working time is about 15 minutes or less and more preferably about 12 minutes or less.

[0047] The adhesive composition is preferably used to bond glass or plastic coated with an abrasion resistant coating, to other substrates such as metal or plastics. In a preferred embodiment the first substrate is a glass, or plastic coated with an abrasion resistant coating, window and the second substrate is a window frame. In another preferred embodiment the first substrate is a glass, or plastic coated with an abrasion resistant coating, window and the second substrate is a window frame of an automobile. Preferably, the glass window is cleaned and has a glass primer applied to the area to which the adhesive is to be bonded. The plastic coated with an abrasion resistant coating can be any plastic which is clear, such as polycarbonate, acrylics, hydrogenated polystyrene or hydrogenated styrene conjugated diene block copolymers having greater than 50 percent styrene content. The coating can comprise any coating which is abrasion resistant such as a polysiloxane coating. Preferably, the coating has an ultraviolet pigmented light blocking additive. Preferably, the glass or plastic window has an opaque coating disposed in the region to be contacted with the adhesive to block UV light from reaching the adhesive.

[0048] In a preferred embodiment the composition of the invention is used to replace windows in structures or vehicles and most preferably in vehicles. The first step is removal of the previous window. This can be achieved by cutting the bead of the adhesive holding the old window in place and then removing the old window. Thereafter the new window is cleaned and primed. The old adhesive that is located on the window flange can be removed, although it is not necessary and in most cases it is left in place. The window flange is preferably primed with a paint primer. The adhesive is applied in a bead to the periphery of the window located such that it will contact the window flange when placed in the vehicle. The window with the adhesive located thereon is then placed into the flange with the adhesive located between the window and the flange. The adhesive bead is a continuous bead that functions to seal the junction between the window and the window flange. A continuous bead of adhesive is a bead that is located such that the bead connects at each end to form a continuous seal between the window and the flange when contacted. Thereafter the adhesive is allowed to cure.

[0049] In another embodiment the compositions of the invention can be used to bond modular components together.



Examples of modular components include vehicle modules, such as door, window or body.

[0050] Viscosities as described herein are determined according to the procedure disclosed in Bhat, U.S. Pat. No. 5,922,809 at column 12 lines 38 to 49, incorporated herein by reference.

[0051] Molecular weights as described herein are determined according to the following to the procedure disclosed in Bhat U.S. Pat. No. 5,922,809 at column 12 lines 50 to 64, incorporated herein by reference.

[0052] In reference to polyurethane prepolymers, average isocyanate functionality is determined according to the procedure disclosed in Bhat, U.S. Pat. No. 5,922,809 at column 12 lines 65 to Column 13, line 26, incorporated herein by reference.

#### Illustrative Embodiments of the Invention

[0053] The following examples are provided to illustrate the invention, but are not intended to limit the scope thereof. All parts and percentages are by weight unless otherwise indicated.

#### Preparation of Polyether Prepolymer

[0054] A polyether polyurethane prepolymer was prepared by mixing 980 g of a polyoxypropylene diol having an average molecular weight of 2,000 with 595 g of a polyoxypropylene triol having an average molecular weight of 4500 and 455 g of a styrene acrylonitrile dispersed polyoxypropylene triol with an average molecular weight of 5400. Mixing was carried out in a reactor by heating the mixture to 48° C. 320 g of diphenylmethane-4,4'-diisocyanate and 0.17 g of stannous octoate were added to the mixture. The whole mixture was then reacted for one hour at 80° C. Finally, 1120 g of a dialkyl phthalate plasticizer was added to the mixture and the mixing was continued for one hour.

#### Preparation of Polyester Prepolymer 1

[0055] A polyester polyurethane prepolymer was prepared by charging 150 g of diphenylmethane-4,4'-diisocyanate (MDI) to a reaction vessel and heating to 48° C. Then 850 g of molten linear polyester diol (Dynacoll 7360) was slowly added and allowed to react for thirty minutes with a maximum allowable temperature of 88° C.

#### Preparation of Polyester Prepolymer 2

[0056] A polyester polyurethane prepolymer was prepared by charging 175 g of a dialkyl phthalate plasticizer to a reaction vessel and heating to 48° C. To this 108 g of diphenylmethane-4,4'-diisocyanate was added and mixed to insure that the MDI was molten. Then 717 g of molten linear copolyester diol (Dynacoll 7330) was slowly added and allowed to react for thirty minutes. The maximum temperature allowed is 88° C. The polyester used demonstrated a melting point of 80-85° C.

#### Compounding of Adhesives

[0057] All of the adhesives were made according to the following compounding procedure using the raw material percentages listed in Table 1 below. The only change between the different formulations is the compounding temperature. Adhesives without the polyester prepolymers

were compounded at room temperature; otherwise, the mixing vessel was heated to 55° C. for the polyester polyurethane prepolymers.

[0058] The mixing vessel was heated to the desired temp if the polyester polyurethane prepolymer were used. The appropriate amounts of polyether prepolymer and multifunctional isocyanate (Desmodur® N3300 or PAPI™ 2020) were charged to the vessel and degassed under vacuum for 15 minutes. The polyester polyurethane prepolymers were then added and the materials were degassed under vacuum for an additional 15 minutes. The carbon black and clay fillers were added and mixed for five minutes at slow speed under vacuum until the fillers were sufficiently wetted by the prepolymers. The mixing speed was increased and the fillers dispersed for 20 minutes. Finally, the dimorpholino diethyl ether and bismuth octoate catalysts and the N-methyl pyrrolidone were added to the mixing vessel and the mixture was mixed under vacuum for an additional 10 minutes.

#### Testing Procedures

[0059] G-Modulus: The shear modulus of the fully cured adhesives was determined by Instron at 10 percent and 20 percent strain. A lap shear sample was made with 5 mm×10 mm×1 mm dimension and fully cured prior to testing.

[0060] Storage Modulus (Green Strength) at Short Cure Times: The storage modulus of the adhesives was determined at short cure times through the use of a Dynamic Stress Rheometer (DSR). The DSR was programmed to apply a stress at a frequency of five rad/sec using 8 mm parallel plate geometry with a plate separation height of 1 mm. The adhesive sample was applied to the plates and the storage modulus was recorded every 2 minutes for the first two hours after dispensing at the indicated temperatures.

[0061] High Temperature Sag Test: An 8 mm by 12 mm isosceles triangle bead of the adhesive is dispensed with a standard caulking gun onto a surface held at a 60° angle from horizontal. Acceptable performance is a minimal deflection of the bead tip; no deflection or change in shape is preferred. The test used was conducted on the adhesives after aging for 10 hours at 80° C. The adhesives were dispensed while still hot at 80° C. and the amount of sag was recorded as the number of mm of deflection of the bead tip.

[0062] Press Flow Viscosity: The press flow viscosity is determined as the time (seconds) required to extrude 20 grams of adhesive through a capillary. The width of the capillary is fixed at 0.203 in (5.1 mm) and the applied pressure is 80 psi (5.5×10<sup>5</sup> Pa). Unless otherwise noted, all press flow viscosity values were determined at 23±1° C.

#### Results

[0063] The dielectric constant of the fully cured adhesive was determined by using a Hewlett Packard 4396A Network/Spectrum Analyzer PC, as per DIN 53482 test.

[0064] Table 1 describes the formulations tested and the results.

[0065] The tested adhesives can be dispensed at room temperature (typically demonstrating a press flow viscosity range of between 20 and 50 seconds for materials that will be applied with a caulking gun).

TABLE 1

	Example	
	1*	2
Polyether based prepolymer containing 30 percent plasticizer	66	58.86
Desmodur ® N3300 polyfunctional isocyanate	1.5	0
PAPI ® 20 polyfunctional isocyanate	0	1.3
DMDEE	0.34	0.34
Bismuth Octoate	0.2	0.2
M-Pyrol	0.3	0.3
Clay	10.66	21.5
Raven 790 Standard Carbon Black	0	16
Raven 1040 Nonconductive Carbon Black	16	0
Polyester based prepolymer 1	3	1.5
Polyester based prepolymer 2	2	0
Total	100	100
G-Modulus (MPa)	2.21	2.19
Dielectric Constant	11	12.5
Rheology (80° C. hot sag)	1 mm	0
Green		
Strength G' (pa) by DSR @23°		

Time (hour)

0	3.45E+04	1.83E+05
0.5	5.46E+04	5.38E+05
1	8.70E+04	9.32E+05
1.5	1.35E+05	1.38E+06
2	1.99E+05	1.78E+06

\*Comparative Example

DMDEE is dimorpholino diethyl ether

[0066] Table 2 Illustrates formulations made as described by Example 2 above with different carbon black formulations.

TABLE 2

Example	Carbon Black	Dielectric Constant	Rheology (80° C. hot sag)
3	Raven 790	12.48	0
4	Standard Cabot Carbon Black	10.83	0
5	Standard Cabot Carbon Black	12.11	0
6	Printex 30	13.06	0
7	Raven 652A	10.2	0
8	Raven 450	5.45	0
9	Raven 500	8.23	0
10	Raven 430	6.19	0
11	Raven 420	7.12	4 mm
12	Raven 410	5.36	4 mm

[0067] The sag performance of examples 3-10 is excellent, the carbons used had the properties of Oil Absorption Number (OAN) of 60 or greater, and surface area of 31 m<sup>2</sup>/g or greater. The low conductivity requirement is demonstrated by each of these adhesives by demonstrating a dielectric constant of less than 15.

[0068] Table 3 demonstrates dielectric constant versus carbon black levels used. Cabot Carbon was used in Examples 13-19. The data shows that the dielectric constant increases with the increased amount of carbon usage. The adhesives meet low conductive specification when conductive carbon is used at 20 percent or less and demonstrate excellent rheology properties. FIG. 1 presents the data of Table 3 in graphic form which graphs the percent carbon black verses the dielectric constant.

Example	Carbon Black %	Dielectric Constant	G-Modulus (MPa) @ 10%	Rheology (80° C. hot sag)
13	12	8.7	2.12	0
14	14	9.78	2.2	0
15	16	10.2	2.4	0
16	18	11.34	2.6	0
17	20	14.16	2.69	0
18*	22	15.57	2.94	0
19*	24	20.48	3.14	0

Comparative

What is claimed is:

1. A composition comprising

a) one or more isocyanate functional polyether based prepolymers containing one or more organic based polymers dispersed therein;

b) one or more isocyanate functional polyester based prepolymers which is solid at 23° C.;

c) one or more polyisocyanates having a nominal functionality of about 3 or greater;

d) one or more conductive carbon blacks in an amount such that the composition has a dielectric constant of about 15 or less; and

e) one or more catalysts for the reaction of isocyanate moieties with hydroxyl groups;

wherein the composition demonstrates upon cure a modulus of 2.0 MPa or greater at 25° C. measured according to ASTM D4065; a dielectric constant of about 15 or less; a sag of an uncured sample of less than 2 mm, a press flow viscosity of about 20 to about 50 and a storage modulus of about 5.3×10<sup>5</sup> Pa or greater.

2. A composition according to claim 1 wherein the isocyanate functional polyester prepolymer exhibits a melting point of about 50° C. or greater.

3. A composition according to claim 2 wherein the polyfunctional polyisocyanate is an oligomer or polymer based on hexamethylene diisocyanate or methylene diphenyl diisocyanate.

4. A composition according to claim 3 wherein

a) the one or more isocyanate functional polyether based prepolymers are present in an amount of about 25 to about 55 percent;

b) the one or more isocyanate functional polyester based prepolymers are present in an amount of about 0.5 to about 10 percent;

c) one or more polyfunctional isocyanates are present in an amount of about 1 to about 8 percent;

d) one or more conductive carbon blacks are present in an amount of about 10 to about 18 percent; and

e) one or more catalysts for the reaction of isocyanate moieties with hydroxyl groups are present in an amount of about 0.005 to about 2 percent;

wherein the percentages are based on the weight of the composition.

5. A composition according to claim 4 wherein the organic based polymer is a thermoplastic polymer, a rubber modified thermoplastic polymer or a polyurea.

6. A composition according to claim 5 wherein the organic based polymer is a rubber modified thermoplastic polymer.

7. A method of bonding two or more substrates together which comprises contacting the two or more substrates together with a composition according to claim 1 disposed along at least a portion of the area wherein the substrates are in contact.

8. A method according to claim 7 wherein the isocyanate functional polyester prepolymer exhibits a melting point of about 50° C. or greater.

9. The method of claim 7 wherein the at least one of the substrates is window glass.

10. The method of claim 9 wherein at least one of the other substrates is a building or a vehicle.

11. The method of claim 10 wherein the substrate is a vehicle.

12. A method according to claim 7 wherein the polyfunctional polyisocyanate is an oligomer or polymer based on hexamethylene diisocyanate or methylene diphenyl diisocyanate.

13. A method according to claim 11 wherein

- a) the one or more isocyanate functional polyether based prepolymers are present in an amount of about 25 to about 55 percent;
- b) the one or more isocyanate functional polyester based prepolymers are present in an amount of about 0.5 to about 10 percent;
- c) one or more polyfunctional isocyanates are present in an amount of about 1 to about 8 percent;
- d) one or more conductive carbon blacks are present in an amount of about 10 to about 18 percent; and
- e) one or more catalysts for the reaction of isocyanate moieties with hydroxyl groups are present in an amount of about 0.005 to about 2 percent;

wherein the percentages are based on the weight of the composition.

14. A method of replacing a window of a vehicle comprising

- i) removing the window from the vehicle;
- ii) applying a composition according to claim 1 to a replacement window or to the flange of the vehicle adapted to hold the window into the vehicle;
- iii) contacting the flange of the vehicle and the replacement window with the composition disposed between the replacement window and the flange of the vehicle; and
- iv) allowing the adhesive to cure.

15. The method of claim 14 wherein the vehicle can be safely driven after 60 minutes from installation of the window into the vehicle.

16. A method according to claim 15 wherein

- a) the one or more isocyanate functional polyether based prepolymers are present in an amount of about 25 to about 55 percent;
- b) the one or more isocyanate functional polyester based prepolymers are present in an amount of about 0.5 to about 10 percent;
- c) one or more polyfunctional isocyanates are present in an amount of about 1 to about 8 percent;
- d) one or more conductive carbon blacks are present in an amount of about 10 to about 18 percent; and
- e) one or more catalysts for the reaction of isocyanate moieties with hydroxyl groups are present in an amount of about 0.005 to about 2 percent;

wherein the percentages are based on the weight of the composition.

\* \* \* \* \*

# Showing special grades conductive

## **CARBON BLACK GRADES:**

### *ACTIVE CARBON BLACK:*

**N-220, N234, N-299, N-326, N-330, N-375, N-339, P-245, P-234, P-324**

1. GIVES HIGH WEAR-RESISTANCE, HIGH RESISTANCE TO RUPTURE, TEAR RESISTANCE TO RUBBER, HIGH DYNAMIC HARDNESS AND ELASTIC REBOUND TO RUBBER, HIGH DURABILITY, AND LOW HYSTERESIS.
2. WIDELY USED FOR PRODUCING:  
HIGH QUALITY, PROTECTING RUBBERS AND PROTECTING STOCKS, TRANSPORTER AND CONVEYOR BELTS, BREAKER RUBBER, TECHNICAL SEALS, CABLE COVERS. IT IS ALSO USED FOR PRODUCING HIGH QUALITY RUBBER MOLDED ARTICLES WORKING IN HARD CONDITIONS.

### *MEDIUM ACTIVE CARBON BLACK:*

**N-550, N-650, N-660, P-514**

1. GIVES HIGH ELASTICITY TO RUBBER, MIDDLE HARDNESS
2. HAS HIGH ABILITY FOR FILING, HIGH PROFILING PROPERTIES
3. WIDELY USED FOR PRODUCING:  
TIRES, TUBES, SLEEVES, PROFILING ARTICLES, TECHNICAL SEALS, MOLDED ARTICLES, ROOF COVERINGS, FRAMED AND BREAKER RUBBERS, CABLES, FOOTWEAR

### *CONDUCTIVE GRADES:*

**YM-76, YM-66**

1. HAVE HIGH IODINE AND DBP PROPERTIES  
USED AS FILLER IN CONDUCTIVE RUBBERS (SLEEVES, BELTS, CONVEYERS AND ETC), FOR PROTECTION FROM CORROSION, SEALING, CONDUCTIVE PLASTICS, AND PAINTS. REPLACES ACETYLENE CARBON BLACK AS CONDUCTIVE FILLER FOR POLYMER MATERIALS

### *ACTIVE GRADE - MPC CHANNEL BLACK:*

**K-354 (PELLETS)**

1. HAS A HIGH DISPERSION VALUE, A LOW STRUCTURAL VALUE AND ACTIVE SURFACE.
2. GIVES A GOOD ENHANCING EFFECT, ENSURES AN INTENSIVE BLACK COLOUR OF MATERIALS, RESISTANT TO THE ULTRAVIOLET RADIATION.
3. FIELD OF APPLICATION –  
FOR EXTRA STRONG INDUSTRIAL RUBBER GOODS, FOR MAKING INTENSIVELY BLACK VARNISHES AND COATS, SYNTHETIC FIBERS, PRINTING INK, CARBON PAPER, TYPEWRITER TYPES, RUBBERS, PAINTWORKS MATERIALS, PAINTING OF POLYMERIC MATERIALS, FOR PERFUMERY AND CARBON GRAPHITE MATERIALS.

### *SEMI-ACTIVE GRADES:*

**N-754, N-762, N-772, N-774, P-701**

1. HAVE LOW VALUES OF DISPERSION, STRUCTURAL FEATURES GOOD DYNAMIC PROPERTIES TO RUBBERS
2. ALLOW TO OBTAIN AN OPTIMUM COMBINATION OF HIGH SERVICE PROPERTIES OF RUBBERS WITH A GOOD PROCESSING OF MIXES.
3. FIELD OF APPLICATION –  
FOR FRAMEWORKS, EXTRUDING RUBBER ARTICLES, TUBES, RUBBER ARTICLES SUBJECT TO DYNAMIC LOADS, INDUSTRIAL RUBBER ARTICLES AND MASTERBATCH, PRINTING INK, AS A PIGMENT FOR PLASTIC MATERIALS, UV-STABILIZER FOR POLYOLEFINS.

### *LOW-ACTIVE GRADES:*

**N-990 (GRANULES), N-991 (POWDER), P-803**

1. CHARACTERIZED BY THE MOST LOW VALUES OF SPECIFIC SURFACE AND STRUCTURE INDEX IN COMPARISON WITH OTHER GRADES.
2. ENSURE EXCELLENT TECHNOLOGICAL PROPERTIES OF RUBBER MIXES WHILE PROCESSING, POSSIBILITY TO ATTAIN A HIGH DEGREE OF FILLING OF POLYMERIC COMPOSITIONS THUS MAKING ALL ECONOMIC ADVANTAGES OF THIS GRADE.
3. FIELD OF APPLICATION –  
CONVEYOR BELTS, AS INACTIVE FILLER FOR TIRES, INDUSTRIAL RUBBER GOODS AND CARBON GRAPHITE MATERIALS.

Special grades conductive

## News from Notch Consulting Group

Developments in Carbon Black, Silica, Rubber Chemicals & Tire Cord

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« ASTM Recognizes John Bailey of Concarb  
Evonik Degussa Shuts Akron Technical Center »

### **Continental Carbon Adds Conductive Blacks, Carbon Nanotubes to its Product Line**

By Notch

Continental Carbon Co. has announced on its [website](#) that it has expanded its product line beyond rubber blacks to include both conductive carbon black and carbon nanotubes (CNTs). According to the site, “carbon nanotubes and conductive carbon blacks represent the next generation of raw materials and are used across a wide range of industries ranging from electro-static thermal dissipation and shielding to composite material constructions.”

Notch understands that the carbon nanotube materials are currently being produced in lab quantities. When commercial-scale production is required, the materials most likely will be produced at Concarb’s Sunray, Texas plant, which has an idle pilot plant that would be modified to nanotube production.

Concarb’s position in [carbon nanotubes](#) is based on both internally developed technology as well as a recently announced partnership with a CNT company called Unidym, Inc. On May 12, 2009, [Arrowhead Research Corporation](#), the parent company of Unidym, [announced](#) that it had entered into a partnership whereby Concarb will take over Unidym’s bulk CNT materials business. Under the agreement, Concarb will form a new subsidiary, Continental Carbon Nanotechnologies, Inc. (CCNI), that will exclusively supply Unidym with proprietary electronics-grade CNTs for its core markets and sell high quality CNTs directly into other, non-competing markets.

The transfer of assets will take place in two steps: the first involves the transfer of certain assets related to the materials business, including manufacturing equipment and select inventory; and the second will include the transfer of certain intellectual property and third party agreements necessary for the materials business as well as a supply agreement for CCNI to provide proprietary CNTs to Unidym.

According to the agreement, Unidym will continue to serve the electronics industry, including the touch-screen and LCD markets and, over the longer term, markets such as solar energy and printable electronics. Mark Tilley, CEO of Unidym, stated, “Continental Carbon’s global manufacturing operations and expertise will be an integral component of our go-to-market strategy. We expect that demand from the touch panel, LCD and solar markets will require a significant ramp-up of CNT capacity in the coming years.”

In the press release, Kim Pan, President of Continental Carbon, was quoted as saying, “We are pleased to join forces with Unidym and look forward to expanding the market for CNTs. We will work exclusively with Unidym to support Unidym’s transparent conductive films business, and expect to pursue several applications for these extraordinary materials beyond Unidym’s core markets.”

According to industry sources, Concarb’s new conductive black products are also based on internally developed technology. According to the company, potential applications for its conductive blacks include electrostatic dissipation, conductive polymers, platinum catalyst support, batteries and energy storage, cables, coatings, packaging for IC parts, automotive parts, and cell phones, among others.

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It should be noted that another US carbon black producer, Sid Richardson, also recently expanded its product portfolio with a new line of conductive blacks under the Sidcon tradename. The line consists of three grades (Sidcon 159, 419, and 119) offering varying levels of conductivity and processability. These three grades complement an existing line of 13 specialty grades that the company has developed and introduced over the last three years or so.

Taken together, the Concarb and Sid Richardson developments mean that all of the US carbon black suppliers (rounded out by Cabot, Evonik, and Columbian Chemicals) can now offer conductive grades. Internationally, Indian-based Phillips Carbon Black has also recently expanded into conductive blacks. These moves reflect a desire to expand the customer base beyond tiremakers and producers of industrial rubber goods.

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This entry was posted on May 14, 2009 at 10:19 pm and is filed under Carbon Black. You can follow any responses to this entry through the RSS 2.0 feed. You can leave a response, or trackback from your own site.

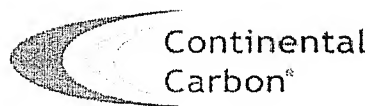
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## Science & Technology

### New Products

- > Carbon Nanotubes
- > Conductive Blacks

## Carbon Blacks, Conductive Blacks & Carbon Nanotubes

A leader in carbon black research, Continental Carbon offers not only rubber grade carbon black products but also sells raw material products in the forms of carbon nanotubes, conductive carbon blacks, and other carbon based products. Carbon nanotubes and conductive carbon blacks represent the next generation of raw materials and are used across a wide range of industries ranging from electro-static thermal dissipation and shielding to composite material constructions. Continental Carbon engages in the manufacture of carbon blacks in 3 locations in the U.S: Sunray, TX, Ponca City, OK, and Phenix City, AL. The Company is committed to delivering quality carbon blacks and providing customers with cutting edge raw materials for applications of carbon nanotube and conductive carbon black technologies.

07.05.09

Continental Carbon Company Settles Class Action Lawsuits, Demonstrates Ongoing Commitment to Corporate and Environmental Stewardship.

05.13.09

John A. Bailey Jr. receives award of merit from ASTM International Committee on carbon black

Carbon Nanotubes

Conductive Blacks

Carbon Blacks

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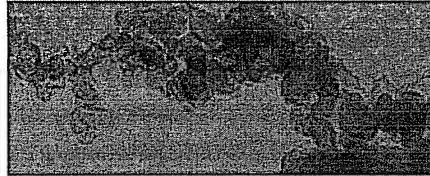
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## Carbon Products

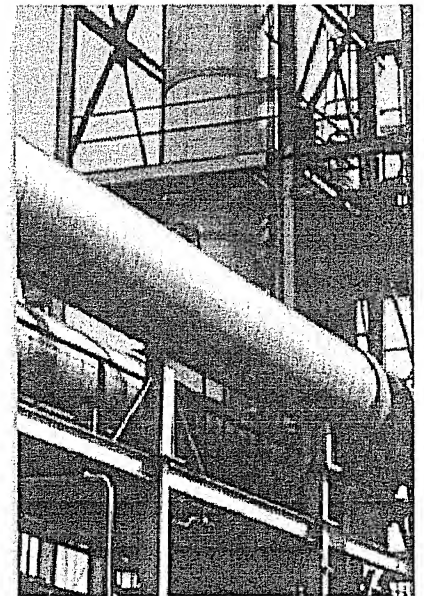
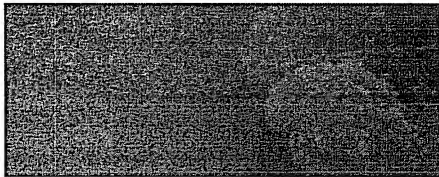
### Carbon Blacks



### Conductive Blacks



### Carbon Nanotubes



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Presented by:

John K. Foster

Special Blacks Division

Cabot Corporation • Billerica, Mass.

at the 2nd International Exhibition of Paint Industry, Suppl. to Vol. 1, No. 1, 1977

#### ABSTRACT

Electrically conductive liquid coatings containing carbon blacks are essential in many applications including primers for automotive plastic parts and coatings for electrostatic dissipation control. This study outlines the important carbon black variables that affect the electrical conductivity of coatings. The analytical properties of the carbon black (e.g. structure, surface area, and volatile content), the carbon black loading, and the dispersion quality all contribute to the conductivity of the cured coating. Proper grade selection and effective incorporation of the carbon black are as important as resin selection in developing a conductive coating.

#### A. INTRODUCTION

Effective conductive coatings are increasingly essential in a broad range of applications. A major segment is that of anti-static protection. This includes products such as: magnetic tape coatings, aircraft paints, spark resistant equipment enamels, etc. Other applications include: primers for electrostatic spraying, coatings for resistive heating, and inks used in printed circuits. Most of these end uses are satisfied by coatings utilizing a conductive filler at various loading levels.

Carbon black is a preferred conductive filler in many cases<sup>1</sup> for several important reasons: (1) readily available, (2) cost effective and, (3) readily dispersed and stabilized in liquid systems.

At high temperatures, usually achieved by incomplete combustion (limited oxygen supply), a hydrocarbon can be dissociated with the rupture of C-H bonds:



The carbon thus formed is the first step in the formation of carbon black. Following this "cracking" stage, carbon and aromatic radicals react to form a planar lattice of hexagonal carbon rings. These "layer planes" then stack together forming bundles several layers thick called crystallites. Subsequently, crystallites combine to form spherical primary particles. These further fuse together forming the primary aggregates, which are characteristic of carbon black [see Figure 1]<sup>2</sup>.

Well over 90 percent of all carbon black is produced today via the Oil Furnace Process. Burning gas in a closed furnace creates a hot flame zone into which an aromatic petroleum distillate is injected. By controlled manipulation of reactor conditions primary particle size, aggregate size, porosity, and surface chemistry can be varied to create a broad range of carbon black properties.

A highly conductive carbon black is the combined effect of these four key properties. Such a black will exhibit fine particle size/high surface area, high structure (highly branched and chained primary aggregates), high porosity and low volatile content (fewer chemisorbed oxygen complexes on the surface). How these properties create enhanced electrical conductivity will be covered in the discussion.

As carbon black loading in a conductive coating is increased, the Surface Resistivity (the reciprocal of surface conductivity) will decrease. This phenomenon is because the quasi-graphitic microstructure of the carbon black makes it much more electrically conductive than any resin. Several percent by weight of carbon black in the dry film is necessary before a significant drop in Surface Resistivity is noted. The amount of carbon black required to obtain a given Surface Resistivity is resin dependent.

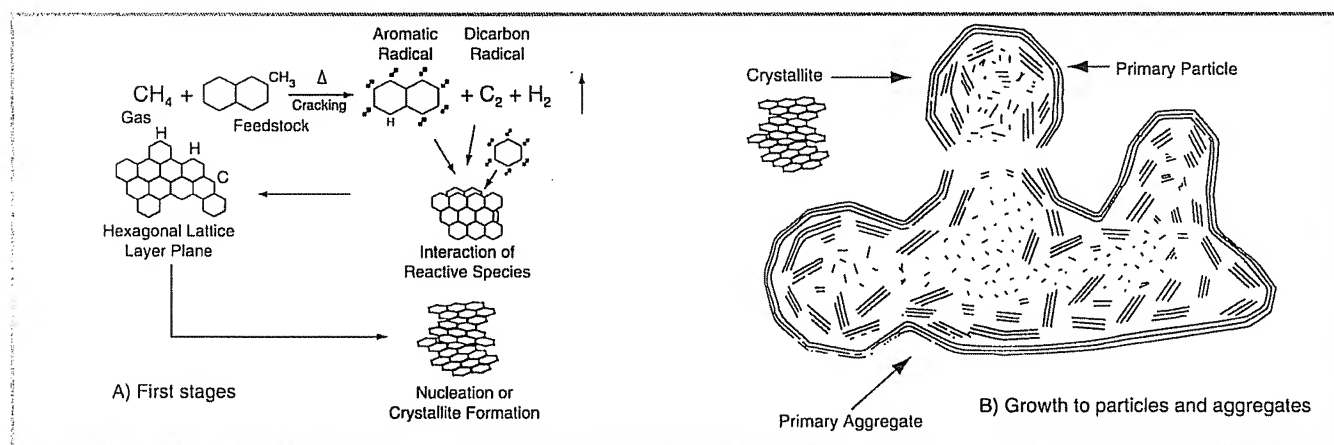


FIGURE 1. FORMATION OF CARBON BLACK

showing must have conductive grade C  
and high enough amount effectively  
dispersed

## B. EXPERIMENTAL

A broad spectrum of carbon black grades, encompassing the range of key properties affecting electrical conductivity, were dispersed in two different coatings formulations. The grades involved, together with their typical properties, are shown in Table I.

TABLE 1.

Grade of Carbon Black	BET Surface Area (m <sup>2</sup> /gram)	DBPA (cc/100 gram)	Volatile
BLACK PEARLS® 1000	343	105	10.0
BLACK PEARLS 880	240	106	1.0
BLACK PEARLS 800	230	68	0.5
VULCAN® XC72	254	176	1.0
VULCAN P	140	115	1.5
BLACK PEARLS L	138	61	5.0
REGAL® 660	112	59	0.5
BLACK PEARLS 570	110	114	1.0
REGAL 400	95	69	3.5
REGAL 330	94	71	1.0
BLACK PEARLS 170	35	122	1.0
CSX*	40	64	1.0

\* CSX refers to a Cabot experimental grade.

Two resin systems, a thermosetting acrylic melamine enamel and an amine cured epoxy, were used in the study in order to compare the relationship of carbon black properties in different systems.

The thermosetting acrylic (TSA) system consisted of dispersing the carbon black at 12 to 34% in 75% solids thermosetting acrylic resin (Hydroxyl Number 86, Acid Number 25) and a 5.2/1.0 n-butanol/methyl-n-amyl ketone solvent blend. The amount of solvent blend used was varied to give final mill paste viscosities of 85 to 110 KU. The carbon black was dispersed for 24 hours in a one-half gallon steel ball mill at 82 rpms. The mill paste was then reduced with additional acrylic resin, hexamethoxymethylmelamine, flow control agent and a solvent blend of 2-ethoxyethyl-acetate, butanol and methyl n-amyl ketone. The reductions were varied to give carbon black loadings of 10 to 35% based on total solids of the system.

The amine-cured epoxy system consisted of dispersing carbon black at 5 to 15% in a bisphenolA/epichlorohydrin based epoxy resin with epoxy equivalent weight of 185-192, silicone, dispersing agent and xylene. The xylene level was varied to give a final mill paste viscosity of 85 to 110 KU. The carbon black was dispersed for 24 hours in a one-half gallon steel ball mill at 82 rpms. The mill paste was reduced with methyl isobutyl ketone and crosslinked with cycloaliphatic amine (equivalent weight 96/H) and amidoamine (equivalent weight 105/H) to give a carbon black loading of 10 to 40% based on total solids of the system.

Finished coatings were applied by drawdown with a Bird Applicator at three wet film thicknesses: 1, 3, and 7 mils.

Surface resistivity measurements were made on all dry films using the device shown in Figure 2.

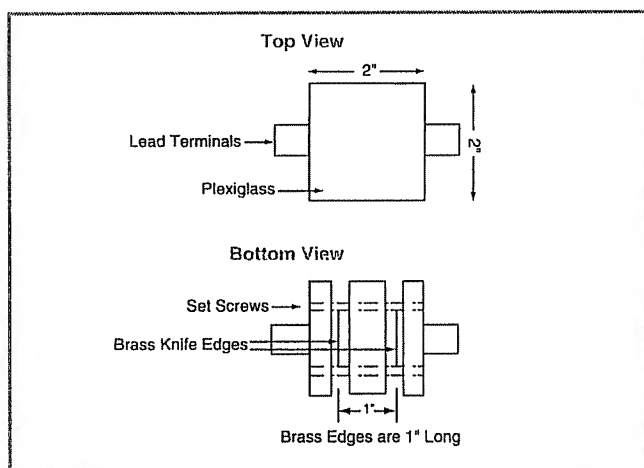


FIGURE 2.  
SURFACE RESISTIVITY DEVICE

## C. RESULTS AND DISCUSSION

Analysis of the data developed in this study clearly shows relationships between the Surface Resistivity of carbon black filled coatings and the key carbon black parameters: dry film loading, particle size/surface area, structure and volatile content. The effects of varying resin system and dry film thickness has likewise been addressed.

### 1. FILM THICKNESS

The thickness of a film has an influence on its measured Surface Resistivity. Figure 3 shows this effect in the TSA system at a 25% carbon black loading by weight.

1. From approximately 1.0 to 3.0 mils wet thickness there is a gradual decrease in Surface Resistivity with respect to increasing film thickness; above 3 mils the change is negligible.

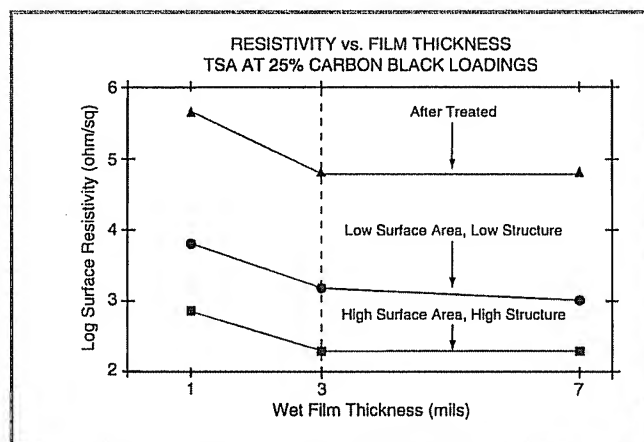


FIGURE 3.

Based on these results, the Surface Resistivity data for the 7 mils thick wet films is used for correlations with carbon black parameters. The effect of film thickness on Surface Resistivity should be considered when designing a coating system.

## 2. CARBON BLACK LOADING

An illustration of the effect of carbon black loading on the Surface Resistivity of coatings is shown in Figures 4 and 5 for the epoxy and TSA systems respectively. These plots clearly show that increased carbon black loading results in lower film Surface Resistivity. Several mechanisms<sup>3,4</sup> have been proposed to account for the effects. The key point is that electron transfer from aggregate to aggregate in the dry film is facilitated by reducing interaggregate gap distances, caused by increased loading, which results in decreased Surface Resistivity.

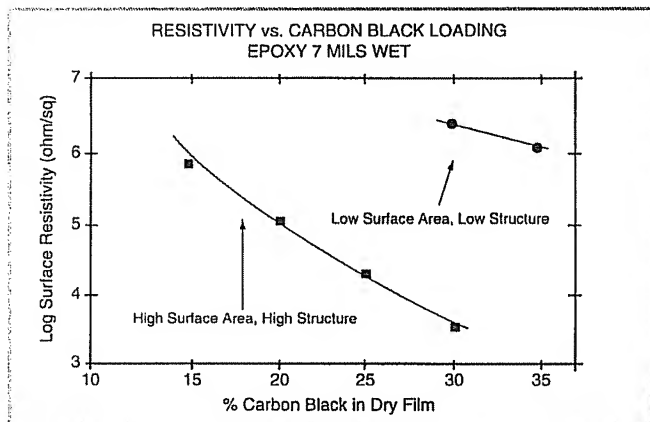


FIGURE 4.

The Surface Resistivity; however, is also dependent on the carbon black properties and the resin system. A high surface area/high structure carbon black, such as Cabot's VULCAN® XC72 carbon black, will result in equal Surface Resistivity at much lower loading than low surface area/low structure carbon blacks. The advantage of a high surface area, high structure carbon black is that at the same Surface Resistivity the lower loading is less likely to cause changes in other physical properties of the coating.

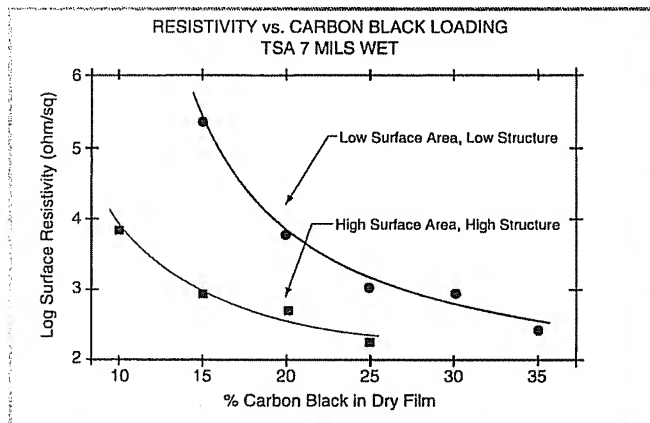


FIGURE 5.

## 3. SURFACE AREA

The surface area of carbon black is determined by measuring both internal and external surface area of the

primary particle. External surface area per unit weight of carbon black increases as the primary particle size decreases. Internal surface area is found in the small pores on the surface of the carbon black. The BET Nitrogen Surface Area is used to measure the total (internal and external) surface area.

As the number of carbon black aggregates per unit weight increases the distance between aggregates decreases. Decreases in interaggregate distance and increases in the occurrence of aggregate to aggregate contact results in a decrease in Surface Resistivity. Both smaller and higher porosity particles yield a greater number of carbon black aggregates per unit weight.

Figures 6 and 7 illustrate the effect of BET Nitrogen Surface Area on Surface Resistivity in both epoxy and TSA systems respectively. As expected, higher surface area carbon blacks have lower Surface Resistivity for a given carbon black loading. However, as the figures indicate, surface area does not completely account for the surface resistivity imparted by different grades of carbon black. Other carbon black properties such as structure and volatile content also play a role in Surface Resistivity and will be discussed subsequently.

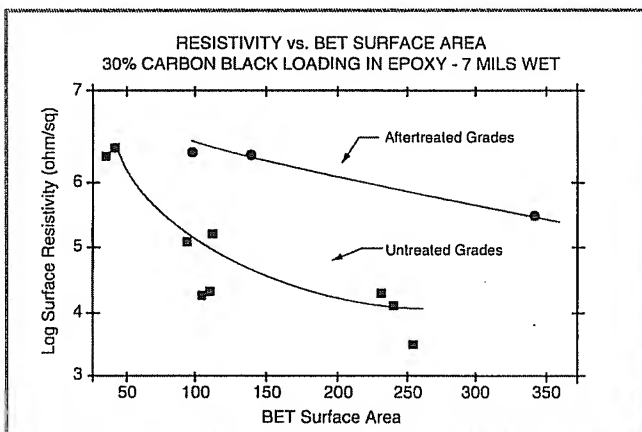


FIGURE 6.

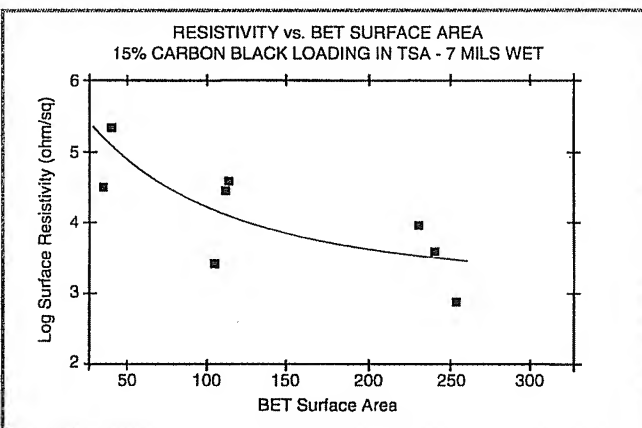


FIGURE 7.

#### 4. STRUCTURE

Structure is a complex property that refers to the morphology of the primary aggregates of carbon black. It is a measure of both the number of primary particles comprising a primary aggregate and the manner in which they are "fused" together. Figure 8 provides a schematic of high structure blacks, characterized by aggregates comprised of many primary particles with considerable "branching" and "chaining", and low structure blacks, characterized by compact aggregates comprised of fewer primary particles. As structure increases, "void" space within an aggregate is created. Dibutyl Phthalate (DBP) or Oil absorbed by these voids is an effective measure of structure.

Structure affects carbon black resistivity in two ways. First, it contributes to the aggregate size. By increasing the number of primary particles per aggregate, and therefore, increasing aggregate weight, higher structure results in fewer carbon black aggregates by weight. However, the dominant effect is believed to be that the increased "branching" and "chaining" of higher structure carbon blacks actually reduces interaggregate gap distance and increases the incidence of aggregate to aggregate contact. The result is lower surface resistivity. The data in Figures 9 and 10 tend to support these statements. In both the epoxy and TSA systems surface resistivity decreases as structure is increased.

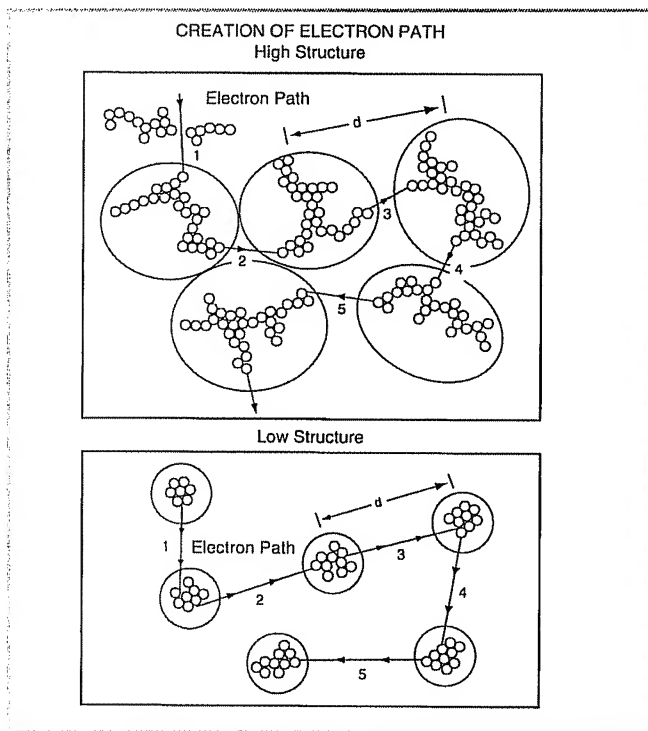


FIGURE 8.

The simulated primary aggregate of a very high-structure black (upper sketch) has gaps (1), (2), (3), (4), and (5) to be considerably smaller than those of a very low structure black (lower sketch) even at a comparable average inter-aggregate distance,  $d$ , as measured center to center. This translates to greater ease of electrons bridging those gaps, and thus increased conductivity.

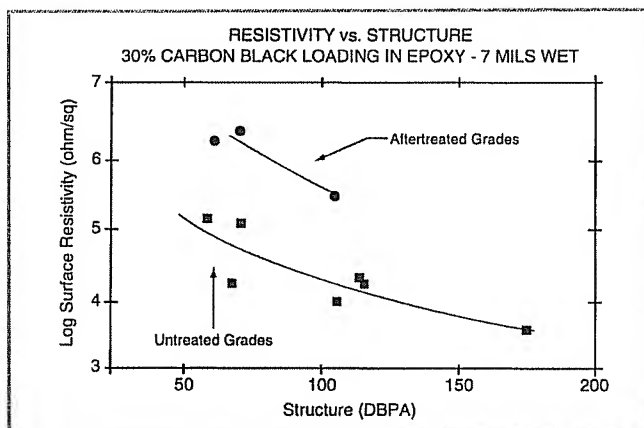


FIGURE 9.

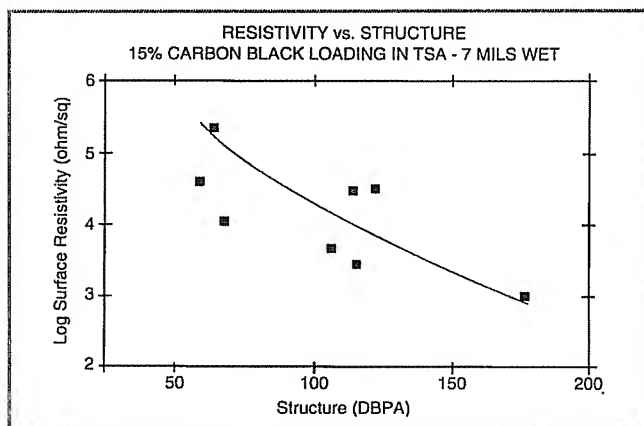


FIGURE 10.

#### 5. VOLATILE CONTENT

The term volatile content, as used in the carbon black industry, refers to chemisorbed oxygen complexes (carboxylic, quinonic, lactonic and hydroxylic groups) present on all carbon blacks to some degree. Since these complexes can be driven off by heating the carbon black to 960°C, the term "volatile" is used. It should be noted that volatile content [see Table I] does not include absorbed moisture.

In the furnace process all carbon blacks are oxidized and acquire a small amount of chemisorbed oxygen complexes. Typically furnace process carbon blacks have volatile contents of 0.5 to 2.0%. To increase the volatile content, carbon blacks are post-oxidized by a variety of techniques. Carbon blacks which have been post-oxidized are referred to throughout this paper as aftertreated.

These polar chemisorbed oxygen complexes increase the wettability of the carbon black surface by polar solvents and resins. Higher volatile content increases the wettability, resulting in a better and faster dispersion of the carbon black in a polar dispersion medium. While higher volatile content is generally desired by the coating producer, it increases Surface Resistivity.

Oxygen complexes act as an electrically insulating layer on the surface of carbon black aggregates so that

aftertreated grades will exhibit higher resistivity than their untreated counterparts.

Figures 11, 12, 13, and 14 demonstrate the effects of volatile content on Surface Resistivity for both epoxy and TSA systems.

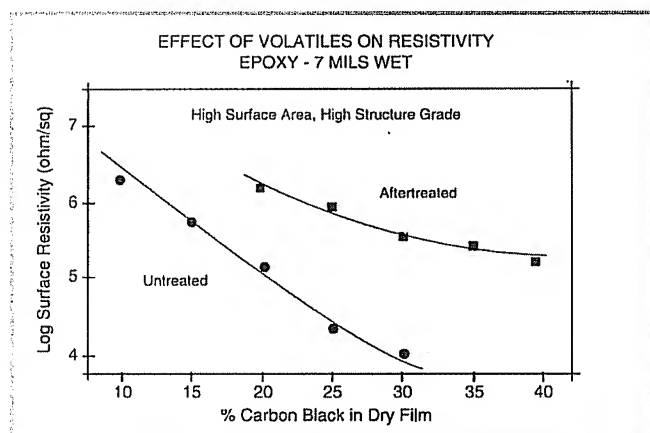


FIGURE 11.

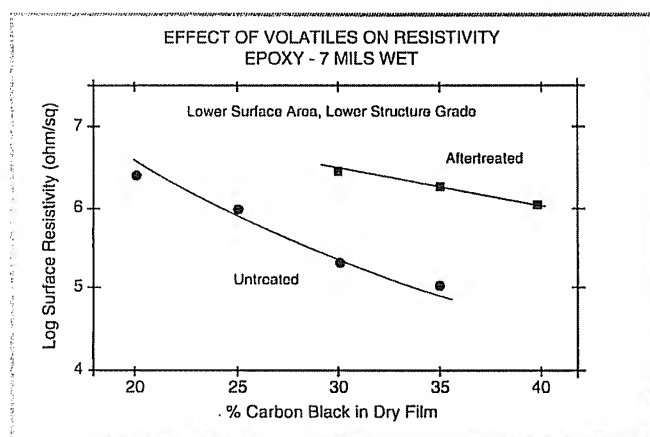


FIGURE 12.

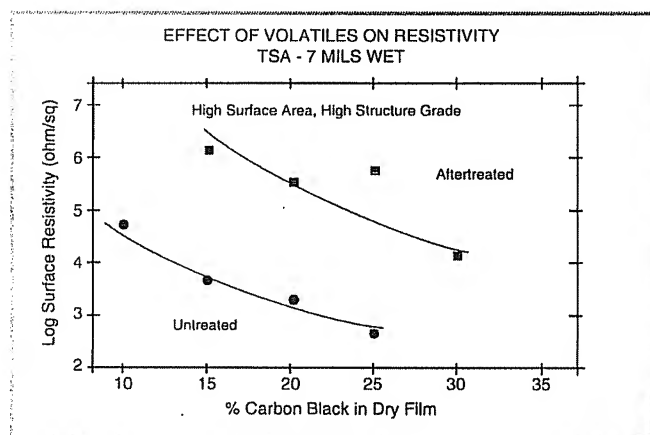


FIGURE 13.

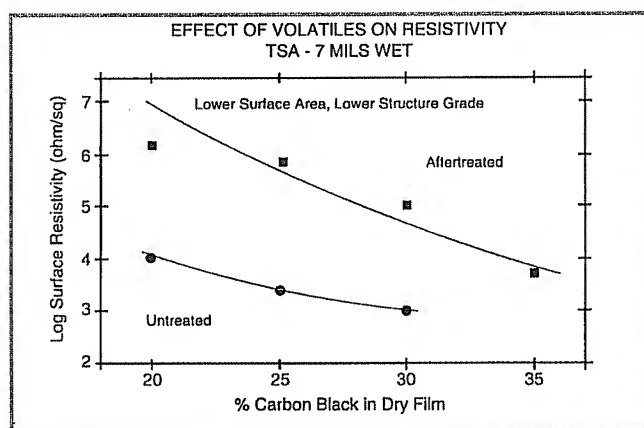


FIGURE 14.

The increase in volatile content creates a dramatic increase in surface resistivity for both high surface area/high structure grades and low surface area/low structure grades of carbon black. For both the epoxy and TSA systems, surface resistivity increases significantly with an increase in volatile content regardless of the morphology of the carbon black.

#### D. FORMULATION GUIDELINES

The properties of the carbon black required to achieve the desired Surface Resistivity will determine the formulator's selection of dispersion technique, the achievable dispersion quality, and the physical properties of the coating. A discussion of the effect of carbon black properties on dispersion and carbon black dispersion techniques is beyond the scope of this paper<sup>5</sup>. However the following generalizations apply when selecting a carbon black:

1. higher surface area carbon blacks are more difficult to disperse
2. higher structure (i.e. higher DBP) carbon blacks are easier to disperse
3. higher volatile content carbon blacks are easier to wet out and disperse

When selecting a dispersion process, the degree of dispersion required in a conductive coating must be considered. For a conductive coating an acceptable degree of dispersion is not necessarily an optimum dispersion. For carbon black to impart conductivity in a film, a network of carbon black aggregates with small interaggregate gap distances and some aggregate to aggregate contact must be achieved. A perfect dispersion where all aggregates are equally spaced and no aggregates contact each other may not be as conductive as a less perfect dispersion. At the other extreme, in a poor carbon black dispersion where many of the aggregates are part of large agglomerates, a network may not exist and the film may not be conductive. Because dispersion quality affects Surface Resistivity and is not easily measured quantitatively, it is critical to closely control the dispersion process so that the same degree of dispersion can be achieved repeatedly.

When formulating an anti-static film, use of a higher surface area carbon black can reduce the carbon black loading necessary to achieve the desired Surface Resistivity. A lower carbon black loading is frequently necessary to maintain other physical properties of the film. Furthermore, in most instances a high surface area carbon black is required to achieve the Surface Resistivity characteristic of a highly conductive film. An effective compromise to an extremely high surface area carbon black is VULCAN® XC72 carbon black which is easy to disperse and provides adequate Surface Resistivity at a carbon black loading that does not cause excessive loss of physical properties.

#### B. CONCLUSIONS

This paper has shown, for two representative resin systems, that surface resistivity of a thin film coating is a function of carbon black properties, carbon black loading, and the resin. In general, high surface area, high structure, low volatile content carbon blacks produce more conductive films. However, high surface area carbon blacks are harder

to disperse and may require additional dispersion time. Furthermore, while low volatile carbon blacks are generally more desirable from a conductive standpoint, high volatile content carbon blacks are generally easier to wet and disperse in polar solvents. The end result is that no single carbon black is the optimum for all applications. The choice is generally determined by the required resin system, the available dispersion technology, and the required surface resistivity.

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**CABOT**

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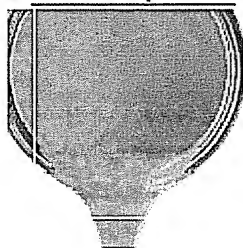
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Chromium pigments



➔ [Material safety data sheets](#)

Elementis Chromium produces a comprehensive range of chrome pigments, used in paints, surface coatings, enamels and in many construction materials. It is also popular for tennis courts and plastics masterbatches.

Manufactured in the US

### G-4099

A light yellowish-green powder which is also available as a higher purity material that meets Food and Drug Administration approval and CTFA specifications for cosmetic use.

To view data sheet [click here](#) (attached)

### G-5099

An intermediate green pigment specifically formulated to expand the range of pigment shades available. This grade is jet milled (micronized) in order to improve pigment characteristics.

To view data sheet [click here](#)

### G-6099

A bluish-green powder that is the darkest shade chromic oxide pigment available with strong tint strength. Also available as a high purity version that meets Food and Drug Administration approval and CTFA specifications for cosmetics.

To view data sheet [click here](#)

### G-8599

A dark green pigment with high infrared reflectance specifically formulated for use in camouflage coatings. It is easily dispersed in and compatible with the resin systems specified for camouflage coatings.

To view data sheet [click here](#)

### GA-4090

A light green powder, specifically developed as a cost-effective, high value product for applications that do not require the level of specifications typically demanded of high performance pigments. GA-4090 is more yellow than GA-6090.

To view data sheet [click here](#)

### GA-6090

A darker green powder than GA-4090, specifically developed as a cost-effective, high value product for applications that do not require the level of specifications typically demanded of high performance pigments.

To view data sheet [click here](#)

### GH-9869 Chrome hydrate

A blue green colour hydrated version of chromic oxide which meets Food and Drug

Administration approval.

To view data sheet [click here](#)

vt

Manufactured in the UK

**M100**

A light yellowish-green shade of the chromic oxide pigment.

To view data sheet [click here](#)

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[Back to chromic oxide page](#)

[↑ Top](#)

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# CHROMIUM OXIDE

## GO-4099

### Cr<sub>2</sub>O<sub>3</sub>

ELEMENTIS  
CHROMIUM

#### PROPERTIES

**Chemical Formula**

Cr<sub>2</sub>O<sub>3</sub>

**Molecular Weight**

152.01

**Appearance**

Green powder

**Pigment Classification**

Pigment Green 17

C.I. 77288

**Particle Shape**

Spheroidal

**Specific Gravity**

5.1

**Solubility in Water**

Insoluble

**Stability in Air**

Completely Stable

**Behavior on Heating**

Completely stable.

Melts at 2300 °C (4172 °F)

**Chemical Characteristics**

Inert except under very vigorous conditions. Reacts slowly in hot concentrated sulfuric acid. Oxidizes in hot perchloric acid. Also reacts with molten alkali at high temperatures, specifically under oxidizing conditions. Not readily reduced by hydrogen or carbon monoxide - high temperatures and pressures are required.

**CAS NUMBER:** 1308-38-9

#### TYPICAL ANALYSIS \*

Chromium as Cr <sub>2</sub> O <sub>3</sub> , %	99.2
Water Soluble Salts, %	0.1
Moisture at 110°C, %	0.1
Specific Surface Area (BET m <sup>2</sup> /gm)	5.0
Retention on 53 micron (325 mesh) screen, %	0.01
pH	7.2

\* Data represent normal production values and may be slightly higher or lower for actual product lots.

## GENERAL APPLICATIONS

Pigment Grade Chromium Oxides or Pure Chromium Oxides, as they are sometimes called, find a variety of applications in paints and coatings, enamels, concrete and other building products, floor coverings, and in other uses where permanence of color is paramount. They are also used as catalysts where chemical composition and physical properties other than color are important.

Characteristics of the Pigment Grade Chromium Oxides, as compared to other green pigments, are their stability and performance. They are unaffected by acids, alkalis, paint vehicles and solvents, and they possess excellent durability against light, harsh weather and extremely high temperatures (such as those used in firing of ceramics).

G-4099 is a light yellowish-green powder and is also available as a higher purity material that meets FDA and CTFA specifications for cosmetic use.

## HEALTH & SAFETY PRECAUTIONS

Chromium (III) Oxide may be regarded as essentially nontoxic. However, excessive exposure may cause irritation to the eyes, skin or lungs. Appropriate precautions should be observed in handling to minimize skin contact and to maintain air concentrations below the TLV of 0.5 mg Cr/m<sup>3</sup>. For detailed information, consult our Material Safety Data Sheet (MSDS), which is available upon request.

## STORAGE

The product should be stored in a dry place.

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Web Site: www.elementis.com

# CHROMIUM OXIDE

## GO-5099

### Cr<sub>2</sub>O<sub>3</sub>



#### PROPERTIES

**Chemical Formula**

Cr<sub>2</sub>O<sub>3</sub>

**Molecular Weight**

152.01

**Appearance**

Green powder

**Pigment Classification**

Pigment Green 17

C.I. 77288

**Particle Shape**

Spheroidal

**Specific Gravity**

5.1

**Solubility in Water**

Insoluble

**Stability in Air**

Completely Stable

**Behavior on Heating**

Completely stable.

Melts at 2300 °C (4172 °F)

**Chemical Characteristics**

Inert except under very vigorous conditions.

Reacts slowly in hot concentrated sulfuric acid. Oxidizes in hot perchloric acid. Also

reacts with molten alkali at high

temperatures, specifically under oxidizing

conditions. Not readily reduced by hydrogen

or carbon monoxide - high temperatures and

pressures are required.

**CAS NUMBER:** 1308-38-9

#### TYPICAL ANALYSIS \*

Chromium as Cr <sub>2</sub> O <sub>3</sub> , %	99.2
Water Soluble Salts, %	0.1
Moisture at 110°C, %	0.1
Oil Absorption, %	12.5
Specific Surface Area (BET m <sup>2</sup> /gm)	3.6
Retention on 53 micron (325 mesh) screen, %	0.02
pH	7

\* Data represent normal production values and may be slightly higher or lower for actual product lots.

## GENERAL APPLICATIONS

Pigment Grade Chromium Oxides or Pure Chromium Oxides, as they are sometimes called, find a variety of applications in paints and coatings, enamels, concrete and other building products, floor coverings, and in other uses where permanence of color is paramount. They are also used as catalysts where chemical composition and physical properties other than color are important.

Characteristics of the Pigment Grade Chromium Oxides, as compared to other green pigments, are their stability and performance. They are unaffected by acids, alkalis, paint vehicles and solvents, and they possess excellent durability against light, harsh weather and extremely high temperatures (such as those used in firing of ceramics).

G-5099 is an intermediate green pigment specifically formulated to expand the range of pigment shades available. This grade is jet milled (micronized) in order to improve pigment characteristics.

## HEALTH & SAFETY PRECAUTIONS

Chromium (III) Oxide may be regarded as essentially nontoxic. However, excessive exposure may cause irritation to the eyes, skin or lungs. Appropriate precautions should be observed in handling to minimize skin contact and to maintain air concentrations below the TLV of 0.5 mg Cr/m<sup>3</sup>. For detailed information, consult our Material Safety Data Sheet (MSDS), which is available upon request.

## STORAGE

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# CHROMIUM OXIDE

## GP-6099

### Cr<sub>2</sub>O<sub>3</sub>



#### PROPERTIES

**Chemical Formula**

Cr<sub>2</sub>O<sub>3</sub>

**Molecular Weight**

152.01

**Appearance**

Green powder

**Pigment Classification**

Pigment Green 17

C.I. 77288

**Particle Shape**

Spheroidal

**Specific Gravity**

5.1

**Solubility in Water**

Insoluble

**Stability in Air**

Completely Stable

**Behavior on Heating**

Completely stable.

Melts at 2300 °C (4172 °F)

**Chemical Characteristics**

Inert except under very vigorous conditions. Reacts slowly in hot concentrated sulfuric acid. Oxidizes in hot perchloric acid. Also reacts with molten alkali at high temperatures, specifically under oxidizing conditions. Not readily reduced by hydrogen or carbon monoxide - high temperatures and pressures are required.

**CAS NUMBER:** 1308-38-9

#### TYPICAL ANALYSIS \*

Chromium as Cr <sub>2</sub> O <sub>3</sub> , %	99.3
Water Soluble Salts, %	0.1
Moisture at 110°C, %	0.1
Specific Surface Area (BET m <sup>2</sup> /gm)	3.1
Retention on 53 micron (325 mesh) screen, %	0.05
pH	7.5

\* Data represent normal production values and may be slightly higher or lower for actual product lots.

## GENERAL APPLICATIONS

Pigment Grade Chromium Oxides or Pure Chromium Oxides, as they are sometimes called, find a variety of applications in paints and coatings, enamels, concrete and other building products, floor coverings, and in other uses where permanence of color is paramount. They are also used as catalysts where chemical composition and physical properties other than color are important.

Characteristics of the Pigment Grade Chromium Oxides, as compared to other green pigments, are their stability and performance. They are unaffected by acids, alkalis, paint vehicles and solvents, and they possess excellent durability against light, harsh weather and extremely high temperatures (such as those used in firing of ceramics).

G-6099 is a bluish-green powder and is also available as high purity material that meets FDA and CTFA specifications for cosmetic use.

## HEALTH & SAFETY PRECAUTIONS

Chromium (III) Oxide may be regarded as essentially nontoxic. However, excessive exposure may cause irritation to the eyes, skin or lungs. Appropriate precautions should be observed in handling to minimize skin contact and to maintain air concentrations below the TLV of 0.5 mg Cr/m<sup>3</sup>. For detailed information, consult our Material Safety Data Sheet (MSDS), which is available upon request.

## STORAGE

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# CHROMIUM OXIDE

## GO-8599

### Cr<sub>2</sub>O<sub>3</sub>



#### PROPERTIES

**Chemical Formula**

Cr<sub>2</sub>O<sub>3</sub>

**Molecular Weight**

152.01

**Appearance**

Green powder

**Pigment Classification**

Pigment Green 17

C.I. 77288

**Particle Shape**

Spheroidal

**Specific Gravity**

5.1

**Solubility in Water**

Insoluble

**Stability in Air**

Completely Stable

**Behavior on Heating**

Completely stable.

Melts at 2300 °C (4172 °F)

**Chemical Characteristics**

Inert except under very vigorous conditions.

Reacts slowly in hot concentrated sulfuric acid. Oxidizes in hot perchloric acid. Also

reacts with molten alkali at high

temperatures, specifically under oxidizing

conditions. Not readily reduced by hydrogen

or carbon monoxide - high temperatures and

pressures are required.

**CAS NUMBER:** 1308-38-9

#### TYPICAL ANALYSIS \*

Chromium as Cr <sub>2</sub> O <sub>3</sub> , %	99.6
Water Soluble Salts, %	0.05
Moisture at 110°C, %	0.02
Specific Surface Area (BET m <sup>2</sup> /gm)	1.0
Retention on 53 micron (325 mesh) screen, %	0.04
Average Particle Size, microns	2.5
pH	6.9

\* Data represent normal production values and may be slightly higher or lower for actual product lots.

## GENERAL APPLICATIONS

Pigment Grade Chromium Oxides or Pure Chromium Oxides, as they are sometimes called, find a variety of applications in paints and coatings, enamels, concrete and other building products, floor coverings, and in other uses where permanence of color is paramount. They are also used as catalysts where chemical composition and physical properties other than color are important.

Characteristics of the Pigment Grade Chromium Oxides, as compared to other green pigments, are their stability and performance. They are unaffected by acids, alkalis, paint vehicles and solvents, and they possess excellent durability against light, harsh weather and extremely high temperatures (such as those used in firing of ceramics).

G-8599 is a dark green pigment with high infrared reflectance specifically formulated for use in camouflage coatings. When combined with cobalt bearing mixed metal oxides, it provides the basis for practical, cost-efficient pigments to meet military specification coatings. G-8599 is easily dispersed in and compatible with the resin systems specified for camouflage coatings.

## HEALTH & SAFETY PRECAUTIONS

Chromium (III) Oxide may be regarded as essentially nontoxic. However, excessive exposure may cause irritation to the eyes, skin or lungs. Appropriate precautions should be observed in handling to minimize skin contact and to maintain air concentrations below the TLV of 0.5 mg Cr/m<sup>3</sup>. For detailed information, consult our Material Safety Data Sheet (MSDS), which is available upon request.

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# CHROMIUM OXIDE

## GO-8599

### Cr<sub>2</sub>O<sub>3</sub>



#### PROPERTIES

**Chemical Formula**

Cr<sub>2</sub>O<sub>3</sub>

**Molecular Weight**

152.01

**Appearance**

Green powder

**Pigment Classification**

Pigment Green 17

C.I. 77288

**Particle Shape**

Spheroidal

**Specific Gravity**

5.1

**Solubility in Water**

Insoluble

**Stability in Air**

Completely Stable

**Behavior on Heating**

Completely stable.

Melts at 2300 °C (4172 °F)

**Chemical Characteristics**

Inert except under very vigorous conditions.

Reacts slowly in hot concentrated sulfuric acid. Oxidizes in hot perchloric acid. Also

reacts with molten alkali at high

temperatures, specifically under oxidizing

conditions. Not readily reduced by hydrogen

or carbon monoxide - high temperatures and

pressures are required.

**CAS NUMBER:** 1308-38-9

#### TYPICAL ANALYSIS \*

Chromium as Cr <sub>2</sub> O <sub>3</sub> , %	99.6
Water Soluble Salts, %	0.05
Moisture at 110°C, %	0.02
Specific Surface Area (BET m <sup>2</sup> /gm)	1.0
Retention on 53 micron (325 mesh) screen, %	0.04
Average Particle Size, microns	2.5
pH	6.9

\* Data represent normal production values and may be slightly higher or lower for actual product lots.

## GENERAL APPLICATIONS

Pigment Grade Chromium Oxides or Pure Chromium Oxides, as they are sometimes called, find a variety of applications in paints and coatings, enamels, concrete and other building products, floor coverings, and in other uses where permanence of color is paramount. They are also used as catalysts where chemical composition and physical properties other than color are important.

Characteristics of the Pigment Grade Chromium Oxides, as compared to other green pigments, are their stability and performance. They are unaffected by acids, alkalis, paint vehicles and solvents, and they possess excellent durability against light, harsh weather and extremely high temperatures (such as those used in firing of ceramics).

G-8599 is a dark green pigment with high infrared reflectance specifically formulated for use in camouflage coatings. When combined with cobalt bearing mixed metal oxides, it provides the basis for practical, cost-efficient pigments to meet military specification coatings. G-8599 is easily dispersed in and compatible with the resin systems specified for camouflage coatings.

## HEALTH & SAFETY PRECAUTIONS

Chromium (III) Oxide may be regarded as essentially nontoxic. However, excessive exposure may cause irritation to the eyes, skin or lungs. Appropriate precautions should be observed in handling to minimize skin contact and to maintain air concentrations below the TLV of 0.5 mg Cr/m<sup>3</sup>. For detailed information, consult our Material Safety Data Sheet (MSDS), which is available upon request.

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# CHROMIUM OXIDE PIGMENT GRADES GA-4090, GA-6090



## PROPERTIES

### Chemical Formula

$\text{Cr}_2\text{O}_3$

### Molecular Weight

152.01

### Appearance

Green Powder

### Pigment Classification

Pigment Green 17

C.I. 77288

### Particle Shape

Spheroidal

### Specific Gravity

5.1

### Solubility in Water

Insoluble

### Stability in Air

Completely Stable

### Behavior on Heating

Completely stable.

Melts at 2300 °C (4172 °F)

### Chemical Characteristics

Inert except under very vigorous conditions.

Reacts slowly in hot concentrated sulfuric acid. Oxidizes in hot perchloric acid. Also

reacts with molten alkali at high temperatures, specifically under oxidizing

conditions. Not readily reduced by hydrogen or carbon monoxide - high temperatures and

pressures are required.

**CAS NUMBER:** 1308-38-9

## TYPICAL ANALYSIS \*

	GA-4090	GA-6090
Chromium as $\text{Cr}_2\text{O}_3$ , %	98.0	98.0
Water Soluble Salts, %	< 0.5	< 0.5
Specific Surface Area, BET $\text{m}^2/\text{gm}$	5.5	3.0
pH	7.2	7.5
Average Particle Size, microns	0.6	0.8
Retention on 325 mesh screen, %	< 0.2	< 0.2

\* Data represent normal production values and may be slightly higher or lower for actual product lots.

## GENERAL APPLICATIONS

Chromium Oxides find a variety of applications as pigments in paints and coatings, enamels, concrete and other building products, floor coverings, and in other color applications where permanence of color is paramount. Other applications include catalysts, abrasives, polishing media, and refractories where chemical composition and physical properties other than color are important.

Characteristics of Chromium Oxides, as compared to other green pigments, include their stability, opacity, and high value performance. They are unaffected by acids, alkalis, paint vehicles, and solvents, and they possess excellent durability against light, harsh weather, and extremely high temperatures, including those used to fire ceramics.

Elementis Chromium has designed Chromium Oxide grades GA-4090 and GA-6090 as cost effective, high value products. These grades find application in architectural and other applications that do not require the level of specifications typically demanded of high performance pigments. GA-4090 is light green in color, whereas GA-6090 is a darker green. GA-4090 is also more yellow than GA-6090.

## HEALTH & SAFETY PRECAUTIONS

Chromium (III) Oxide may be regarded as essentially nontoxic. Excessive exposure, however, may cause irritation to the eyes, skin, or lungs. Appropriate precautions should be observed in handling to minimize skin contact and to maintain air concentrations below the TLV of 0.5 mg/m<sup>3</sup> Cr. For detailed information, consult our Material Safety Data Sheet (MSDS), which is available upon request.

## STORAGE

The product should be stored in a dry place.

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# CHROMIUM OXIDE PIGMENT GRADES GA-4090, GA-6090



## PROPERTIES

**Chemical Formula**  
 $\text{Cr}_2\text{O}_3$

**Molecular Weight**  
152.01

**Appearance**  
Green Powder

**Pigment Classification**  
Pigment Green 17  
C.I. 77288

**Particle Shape**  
Spheroidal

**Specific Gravity**  
5.1

**Solubility in Water**  
Insoluble

**Stability in Air**  
Completely Stable

**Behavior on Heating**  
Completely stable.  
Melts at 2300 °C (4172 °F)

**Chemical Characteristics**  
Inert except under very vigorous conditions. Reacts slowly in hot concentrated sulfuric acid. Oxidizes in hot perchloric acid. Also reacts with molten alkali at high temperatures, specifically under oxidizing conditions. Not readily reduced by hydrogen or carbon monoxide - high temperatures and pressures are required.

**CAS NUMBER:** 1308-38-9

## TYPICAL ANALYSIS \*

	GA-4090	GA-6090
Chromium as $\text{Cr}_2\text{O}_3$ , %	98.0	98.0
Water Soluble Salts, %	< 0.5	< 0.5
Specific Surface Area, BET $\text{m}^2/\text{gm}$	5.5	3.0
pH	7.2	7.5
Average Particle Size, microns	0.6	0.8
Retention on 325 mesh screen, %	< 0.2	< 0.2

\* Data represent normal production values and may be slightly higher or lower for actual product lots.

## GENERAL APPLICATIONS

Chromium Oxides find a variety of applications as pigments in paints and coatings, enamels, concrete and other building products, floor coverings, and in other color applications where permanence of color is paramount. Other applications include catalysts, abrasives, polishing media, and refractories where chemical composition and physical properties other than color are important.

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Elementis Chromium has designed Chromium Oxide grades GA-4090 and GA-6090 as cost effective, high value products. These grades find application in architectural and other applications that do not require the level of specifications typically demanded of high performance pigments. GA-4090 is light green in color, whereas GA-6090 is a darker green. GA-4090 is also more yellow than GA-6090.

## HEALTH & SAFETY PRECAUTIONS

Chromium (III) Oxide may be regarded as essentially nontoxic. Excessive exposure, however, may cause irritation to the eyes, skin, or lungs. Appropriate precautions should be observed in handling to minimize skin contact and to maintain air concentrations below the TLV of 0.5 mg/m<sup>3</sup> Cr. For detailed information, consult our Material Safety Data Sheet (MSDS), which is available upon request.

## STORAGE

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# CHROMIUM HYDRATE

## GH-9869

$$\text{Cr}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$$

### PROPERTIES

#### Chemical Formula

$\text{Cr}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$

#### Molecular Weight

188.05

#### Appearance

Blue-Green Powder

#### Pigment Classification

Pigment Green 18

C.I. 77289

#### Particle Shape

Spheroidal

#### Specific Gravity

2.9

#### Solubility in Water

Insoluble

#### Chemical Characteristics

Can react with molten alkali at very high temperatures, specifically under oxidizing conditions.

**CAS NUMBER:** 12001-99-9

### TYPICAL ANALYSIS \*

Chromium as $\text{Cr}_2\text{O}_3$ , %	76
Specific Surface Area (BET $\text{m}^2/\text{gm}$ )	130
Water Soluble Salts, %	1
Retention on 53 micron (325 mesh) screen, %	0.01
Lead, ppm	< 5
Arsenic, ppm	< 0.5
Mercury, ppm	< 0.5
pH	8

\* Data represent normal production values and may be slightly higher or lower for actual product lots.

Product meets specifications of the Food and Drug Administration (FDA, 21 CFR 73.1326) and the Cosmetic, Toiletry and Fragrance Association (CTFA)

## GENERAL APPLICATIONS

Chromium Hydrate GH-9869 is a bright blue-green powder manufactured in the United States solely by Elementis Chromium LP. It is widely used in cosmetics and personal care products.

Chromium Hydrate meets specifications of both the Food and Drug Administration and the Cosmetic Toiletry and Fragrance Association.

Chemically, Chromium Hydrate is a hydrated form of chromium oxide bound with two molecules of water. It is insoluble in water and compatible with many organic materials.

The high surface area of Chromium Hydrate also lends itself to utilization in catalyst applications.

## HEALTH & SAFETY PRECAUTIONS

When handling Chromium Hydrate, care should be taken to avoid generating airborne dust. Proper ventilation should be available to maintain air concentrations below the TLV of 0.5 mg Cr/m<sup>3</sup>. Excessive exposure may cause irritation to the eyes, skin and lungs; therefore, to minimize contact, protective clothing and equipment and good hygiene practices are recommended during handling.

## STORAGE

A dry storage area is recommended.

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# ELEMENTIS

CHROMIUM

## SPECIFICATION AND DATA SHEET

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### CHROMIC OXIDE – M100

#### GENERAL DESCRIPTION AND CHARACTERISTICS

Fine green powder.  
Molecular formula,  $\text{Cr}_2\text{O}_3$ .  
Molecular weight, 151.99.  
EEC No. 215-160-9.  
CAS No. 1308-38-9.  
Melting point  $2300^\circ\text{C}$ .  
True density  $5.22\text{g/cm}^3$ .  
Bulk density approx.  $0.7\text{g/cm}^3$ .  
Insoluble in water and organic solvents.  
Chemically inert, only reacts with strong or oxidising acids and alkalis at elevated temperatures.

#### SPECIFIED CHARACTERISTICS – Standard Grade

		<u>Maximum</u>	<u>Minimum</u>
	% $\text{Cr}_2\text{O}_3$		98.7
	% $\text{H}_2\text{O}$	0.2	
[1]	% $\text{Cr}^{6+}$	0.003	
	% Fe	0.1	
[2]	% $>63\ \mu\text{m}$	0.2	
[2]	% $>425\ \mu\text{m}$	0.0	
	% Soluble Salts	0.2	
[3]	Colour dL vs. Std.	1.4	-1.4

#### Note

- [1] Water soluble expressed as  $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ .  
[2] Wet sieving technique.  
[3] Full shade dispersion in nitrocellulose lacquer.

A DETAILED SAFETY DATA SHEET IS AVAILABLE ON REQUEST